1984

The effect of mould size on the exothermic reaction and compressive strength of epoxy resin chocks.

Stalnaker, John D.

NAVAL POSTGRADUATE SCHOOL
Monterey, California

THESIS

THE EFFECT OF MOULD SIZE ON THE
EXOTHERMIC REACTION AND
COMPRESSIVE STRENGTH OF
EPOXY RESIN CHOCKS

by

John D. Stalnaker

December 1984

Thesis Advisor: D. Salinas

Approved for public release; distribution unlimited.

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) The Effect of Mould Size on the Exothermic Reaction and Compressive Strength of Epoxy Resin Chocks		5. TYPE OF REPORT & PERIOD COVERED Master's Thesis December 1984
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) John D. Stalnaker		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Postgraduate School Monterey, California 93943		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Postgraduate School Monterey, California 93943		12. REPORT DATE December 1984
		13. NUMBER OF PAGES 71
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Chocks, Epoxy Resin, CHOCKFAST ORANGE, Exothermic Reaction, Compressive Strength

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A study was conducted on the effects of mould size on the exothermic reaction that occurs during the curing of epoxy resin chocks. Moulds of CHOCKFAST ORANGE were cast with lateral dimensions of 3 by 3 inches, 6 by 6 inches and 9 by 9 inches, with thicknesses of 1/2, 1 and 1-1/2 inches. Results showed that the degree of exotherm increased with increased chock thickness. The 1/2 inch thick samples showed

very little temperature rise, with the temperature increasing linearly with time. The 1 inch thick samples, beginning with the 6 by 6 by 1 inch sample, and the 1-1/2 inch samples experienced a two stage exothermic reaction, with temperature increasing linearly initially and then exponentially during the second stage. Stress versus strain plots revealed a significant increase in ductility with the increased exotherms, resulting in substantially higher ultimate strengths. Chock specimens experiencing the second stage, exponential temperature rise had ultimate strengths consistently in excess of 20,000 psi, compared to 16,000 to 17,000 psi to those specimens that did not.

Approved for public release; distribution unlimited.

The Effect of Mould Size on the Exothermic Reaction and Compressive Strength of Epoxy Resin Chocks

by

John D. Stalnaker
Lieutenant, United States Navy
B.S., United States Naval Academy, 1978

Submitted in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE IN MECHANICAL ENGINEERING

from the

NAVAL POSTGRADUATE SCHOOL
December 1984

John N. Dyer, Dean of Science and Engineering

ABSTRACT

A study was conducted on the effects of mould size on the exothermic reaction that occurs during the curing of epoxy resin chocks. Moulds of CHOCKFAST ORANGE were cast with lateral dimensions of 3 by 3 inches, 6 by 6 inches and 9 by 9 inches, with thicknesses of 1/2, 1 and 1-1/2 inches. Results showed that the degree of exotherm increased with increased chock thickness. The 1/2 inch thick samples showed very little temperature rise, with the temperature increasing linearly with time. The 1 inch thick samples, beginning with the 6 by 6 by 1 inch sample, and the 1-1/2 inch samples experienced a two stage exothermic reaction, with temperature increasing linearly initially and then exponentially during the second stage. Stress versus strain plots revealed a significant increase in ductility with the increased exotherms, resulting in substantially higher ultimate strengths. Chock specimens experiencing the second stage, exponential temperature rise had ultimate strengths consistently in excess of 20,000 psi, compared to 16,000 to 17,000 psi to those specimens that did not.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

I. INTRODUCTION

A. BACKGROUND

A chock is defined as a wedge or block used for supporting machinery. The past standard chocking process involved the use of steel or cast iron shims which were hand-fitted and machined to obtain the necessary tolerance, a tedious and time consuming process. The fitting of chocks required a trial and error procedure to ensure a proper fit. After installation, there was the possibility of long term creep and fretting of the metal chocks. The entire process required a large expenditure of manhours and money to complete each specific installation. [Ref. 1]

Epoxy resin chocks have been used commercially for over thirty-five years and over the last ten years have gained widespread acceptance throughout the commercial shipbuilding community. The major consideration in the use of epoxy resin chocks by the commercial shipyards are cost and assurance that the material can support the full equipment load. The use of pour-in-place chocks (PIPC) is considered to be cost effective in most applications because:

a) It eliminates the need to fabricate, hand-fit and weld metal chocks.

b) It eliminates the need to machine-mate surfaces of equipment and foundations.

c) It makes securing the correct equipment alignment or tilt simpler.

d) PIPC is used by trained personnel, although not necessarily skilled craftsmen.

e) The pouring process takes only days to complete, saving days or weeks of production time.

f) Long range problems involving fretting and wear are reduced. [Ref. 2]

CHOCKFAST ORANGE pour-in-place chocks have been used in United States Navy ships since the early 1970's on a case by case basis with NAVSEA approval. In the mid 1970's, Litton-Ingalls began using PIPC on new construction projects, such as the DD 963 class and LHA. Although the epoxy chocks supported relatively light loads in most areas, the service experience has been excellent. In the DD 963 class destroyer, the following installations of CHOCKFAST ORANGE were noted: sonar dome to hull, auxiliary machinery, ASROC magazine, radar foundations, and ammunition racks. Recently the USS ELLIOT underwent a major overhaul at Todd Pacific Shipyards and all PIPC installations were found to be in satisfactory condition. [Ref. 3]

CHOCKFAST ORANGE is an epoxy based, pourable compound. It comes in a two part kit consisting of a can of epoxy resin and a plastic bottle of hardener. The two parts are mixed together and poured into a dammed enclosure (Figure 1).

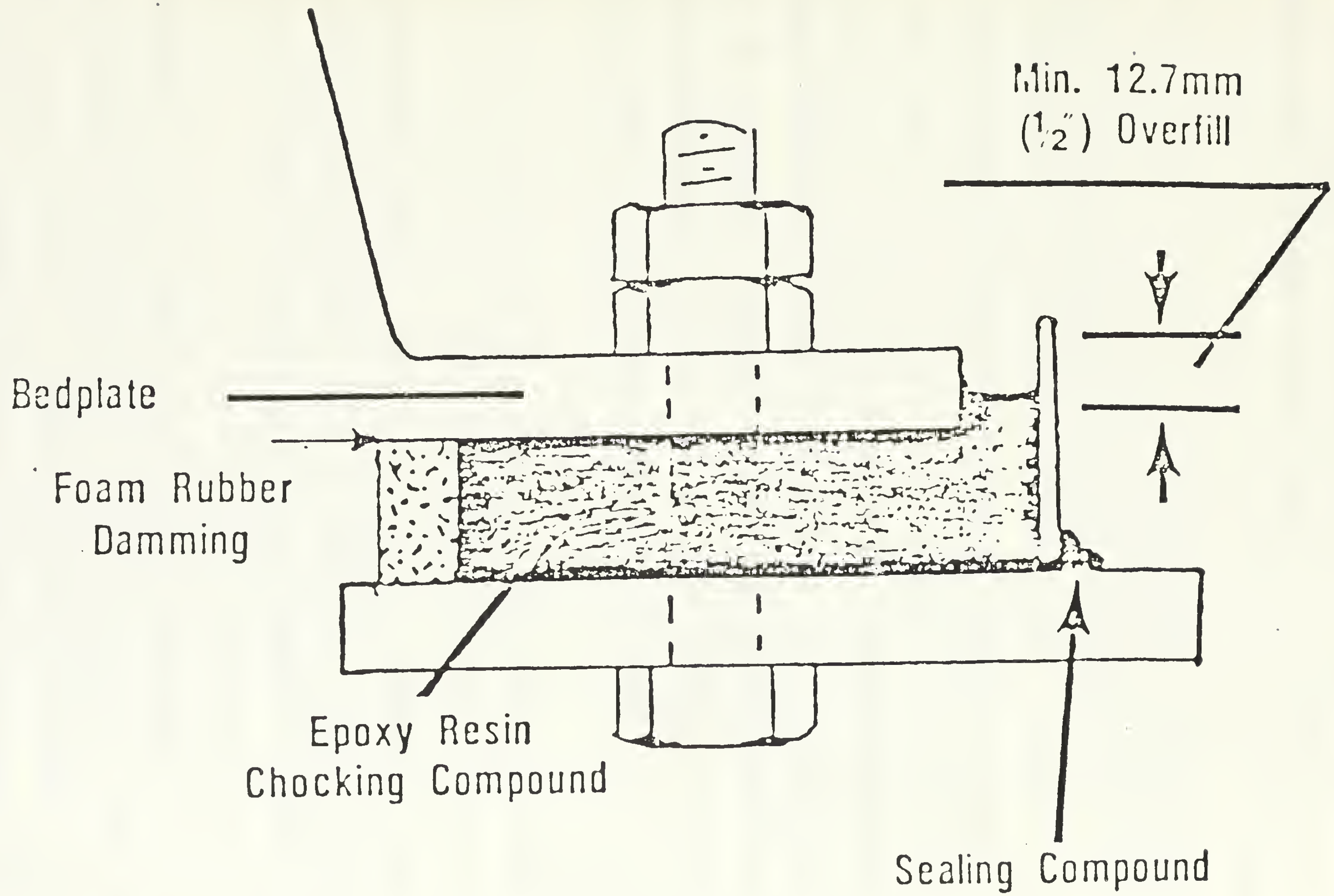

Figure 1. Pour-In-Place Chock (PIPC) [Ref. 4]

The compound cures and solidifies in 12 to 48 hours, dependent on ambient conditions. After the curing process, the dams are removed and the machinery will be supported and properly aligned.

B. STATEMENT OF THE PROBLEM

During the curing process of CHOCKFAST ORANGE an exothermic reaction occurs. The degree of reaction and the maximum temperature achieved varies, dependent on the lateral dimensions and thickness of the mould.

The objective of this study was twofold. First, to vary the dimensions of the moulds and measure the maximum temperature achieved during the curing process. Secondly, to measure the effect of temperature on the ultimate compressive strength, modulus of elasticity, and compressive yield strength of the material.

Chocks were cast with lateral dimensions of 3 by 3 inches, 6 by 6 inches, and 9 by 9 inches, with thicknesses of 1/2, 1, and 1 1/2 inches. From the results, it was determined what effect the mould size had on the exothermic reaction and whether an optimum size and thickness is available to maximize the strength of the chocks.

Two other samples were poured in addition to those mentioned above. Two 6 by 6 by 1 inch samples were poured in different sized moulds to determine if the size of the containment vessel has any effect on the exothermic reaction.

The research is part of an ongoing test program sponsored by the Naval Sea Systems Command (NAVSEA) and the results obtained will be useful in certifying the use of epoxy resin chocks in the installation of main propulsion and auxiliary equipment onboard United States Navy ships. Additionally, the information will be useful to NAVSEA in development of an epoxy chock installation manual.

C. THE NATURE OF EPOXY RESINS AND HARDENER

The term epoxy refers to a chemical group consisting of an oxygen atom bonded with two carbon atoms already united in some other way. The simplest epoxy is a three-numbered ring or α-epoxy. Ethylene oxide is an example of this type and is shown below:

$$\begin{array}{c} O \\ / \quad \backslash \\ CH_2\text{-}CH_2 \end{array}$$

1
Ethylene oxide

The term epoxy resin describes a molecule containing more than one α-epoxy group, that is capable of being transformed from a thermoplastic (uncured) to a thermoset (cured) state. [Ref. 5]

The first commercial epoxy resins consisted of the reaction products of epichlorohydrin and bisphenol A, the reaction giving diglycidyl ether of bisphenol A (DGEBA). [Ref. 6] Although the specific manufacturing process of CHOCKFAST ORANGE was not provided, the major constituent is DGEBA.

The popularity of epoxy resins has increased because of its excellent adhesion, ease of curing, and mechanical strength. The most valuable property is the ability to transform from a liquid state to a usable solid. The conversion is accomplished with the use of a chemically active compound known as a curing agent. Depending on the particular agent used, curing may be accomplished at room temperature with excess heat given off, or may require the application of an external heat source. [Ref. 7] In the case of CHOCKFAST ORANGE, an amine curing agent is used, resulting in an exothermic reaction when curing at room temperature.

The heat of polymerization of an epoxy resin is the amount of heat given off during the curing reaction. In the polymerization reaction, the heat, if given off in a short period of time, will result in a substantial increase in the temperature of the mixture. The temperature increase, referred to as exotherm, may reach 260°C for DGEBA systems cured at room temperature. [Ref. 8]

D. PREVIOUS WORK

Research has been conducted by Industramar Limited, the manufacturer's English distributor on the effect of chock size on the modulus of elasticity. The results of the testing revealed that the modulus of elasticity increased with the chock size. All the tests were conducted at a constant 22.5°C using a Series 1250 Dynamic Materials Testing Instrument. The results are provided in Table I. [Ref. 9]

Table I.

Previous Test Results for Modulus of Elasticity

SAMPLE	MODULUS OF ELASTICITY
1 by 1 by 1 inch	605,800 psi
2 by 2 by 1 inch	700,400 psi
4 by 4 by 1 inch	1,032,472 psi

Additionally, Philadelphia Resins Corporation conducted tests using ASTM D695 standards and a sample size of 0.5 by 0.5 by 1 inch and obtained a value for modulus of elasticity of 550,000 psi.

Lloyds Register of Shipping conducted tests on the exotherm generated during the curing process. The exotherm was measured by insertion of thermocouples into the material after pouring and recorded the temperature using an electronic thermometer. The results are given below in Table II. [Ref. 10]

Table II.

Previous Test Results of Exothermic Reaction

MOULD TYPE	VOLUME (IN 3)	EXOTHERM
Circular, 0.5 in. thick	2.66	30.6 C (87.1 F)
Circular, 1.0 in. thick	5.32	39.6 C (103.3 F)
Circular, 2.0 in. thick	10.64	72.3 C (162.1 F)
Circular, 4.0 in. thick	21.28	99.7 C (211.5 F)
Circular, 1.36 in. thick	11.0	35.3 C (95.5 F)
Circular, 1.36 in. thick	11.0	38.4 C (101.1 F)

II. EXPERIMENTAL APPARATUS AND PROCEDURE

A. MOULD CONSTRUCTION

The first rpoblem faced by the author was designing an appropriate mould. In studying the uses of CHOCKFAST ORANGE and talking with Mr. George Kollock of Philadelphia Resins Corporation, it was determined the best results would be obtained using two steel plates as the mating surfaces and polyurethane or foam rubber as a damming material, since these would most closely represent the in-service environment.

Secondly, there was the problem of mould size. During the course of the study, the size of the moulded samples would cover nine different lateral dimensions or thicknesses. Construction of a different mould for each size would have been too costly in materials and shop man-hours. A total of three moulds were constructed for use during the research.

The final design consisted of two steel plates with dimensions 7 by 7 inches. The plates were secured at each corner with 5/16 inch securing bolts. Cylindrical spacers, which fit over the securing bolts were used to allow for the proper mould thicknesses of 1/2, 1, and 1-1/2 inches. Two such moulds were manufactured and were used to construct the 3 by 3 inch and 6 by 6 inch series of test samples. A third, larger mould of 10 by 17 inches was manufactured and used in constructing the 9 by 9 inch series of test samples and the one

9 by 15 inch test sample, which was originally scheduled to be poured as part of the research.

The damming material decided upon was foam rubber, because of availability and relative ease in cutting the necessary sizes. Several pour attempts were made before arriving at an acceptable damming arrangement. CHOCKFAST ORANGE has great searching power in the liquid state; therefore, it was necessary to overlap the foam rubber sections at the corners and caulk the interface area between the sections. (Fig. 2) Petroleum jelly performed extremely well as a culking agent to stop the flow of CHOCKFAST ORANTE that worked into the cracks and to lubricate the dam surfaces to prevent adhesion during curing.

Secondly, it was necessary to cut the foam rubber thick enough to prevent the chocking material from escaping from between the dams and the steel plates. Several thicknesses were tried before arriving at an acceptable arrangement. The problem was if the foam rubber was not thick enough, the mould would leak and if the foam rubber was too thick, the dams would deform and it would be difficult to pour an accurate sized mould. The best arrangement was cutting the foam rubber to a thickness of 1/2 inch greater than the required thickness of the mould. This compressed the foam rubber enough to resist leaks, but did not deform the foam rubber to the point of effecting mould size.

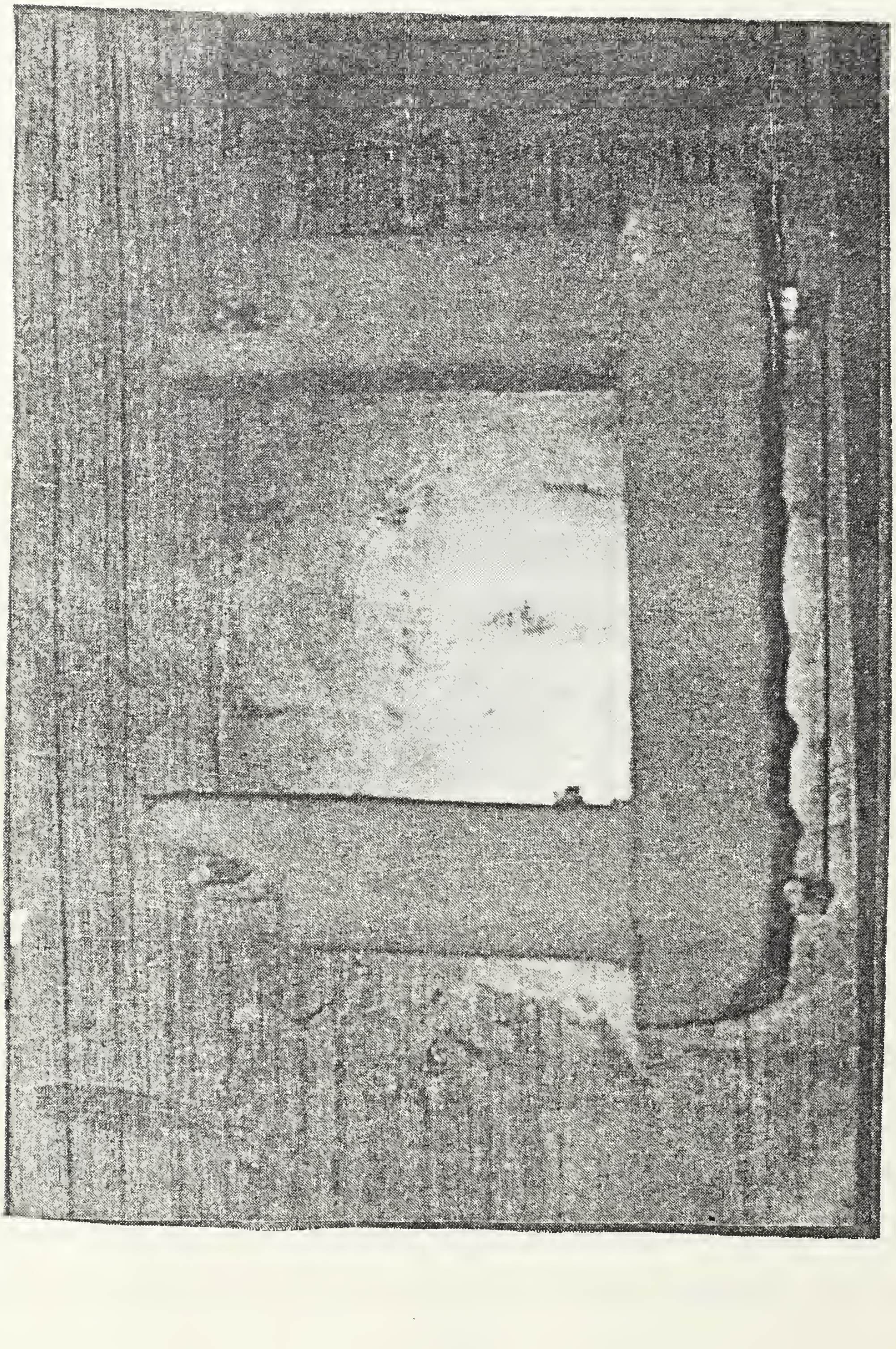

Figure 2. Damming Arrangement

B. TEMPERATURE MEASUREMENT

A continuous time versus temperature recording was made of each test sample poured. Two 24 gauge type-T thermocouples were placed at the center of the mould prior to pouring. The length of the thermocouples was measured, so as to extend to the geometric center of the mould, and the thermocouples were secured to one of the steel plates prior to tightening down the securing bolts. Once the mould was together, the position was checked again to ensure the bead of the thermocouple was precisely at the center. During pouring, the solution was poured slowly at the outside edge of the mould, so the flow would not upset the thermocouple position. This problem was significant in deciding thermocouple size. An initial pour using a smaller size 30 gauge thermocouple failed because the thermocouple was too light and not rigid enough to withstand the flow. Using some sort of securing device at the mould center was considered, but the possible effect on the exothermic was unknown. In an effort to ensure accurate temperature results, a minimum of two pours were done for each thickness of the 3 by 3 inch and 6 by 6 inch series of chocks. This was done to obtain good data correlation. If a large discrepancy in temperature occurred in the results of the two pours, a third pour attempt was made and the erroneous data thrown out.

One of the thermocouples was connected to a Newport digital temperature display, which was monitored continuously

until the maximum curing temperature was reached and temperatures were recorded on a standard data sheet. The second thermocouple was connected through an Omega thermocouple D.C. millivolt amplifier to a Hewlett-Packard type 7132A strip chart recorder, which gave a plot of time versus temperature (Fig. 3). The two thermocouples were checked against each other prior to each pour to ensure the variation in temperature was not greater than $0.1^{\circ}F$.

One major assumption was required in verifying the effect of the exothermic reaction on the compressive strength of the chocks. For the 3 by 3 inch chocks, two samples were needed for each thickness in order to both measure temperature and test compressive strength. Once the mould cured the thermocouples could not be removed, therefore a second mould was needed to be used for compressive testing. The assumption was made that the presence of the thermocouple did not effect the curing and that two samples poured from the same batch, in similar moulds, with the same ambient conditions will respond the same, both in curing and in strength.

C. POURING PROCEDURES

The pouring procedure used throughout the research project was that provided by the manufacturer of CHOCKFAST ORANGE, with one notable exception. The manufacturer recommended using one complete can of epoxy resin and one complete bottle of hardener for each use. The 15 pound can contains 220 cubic

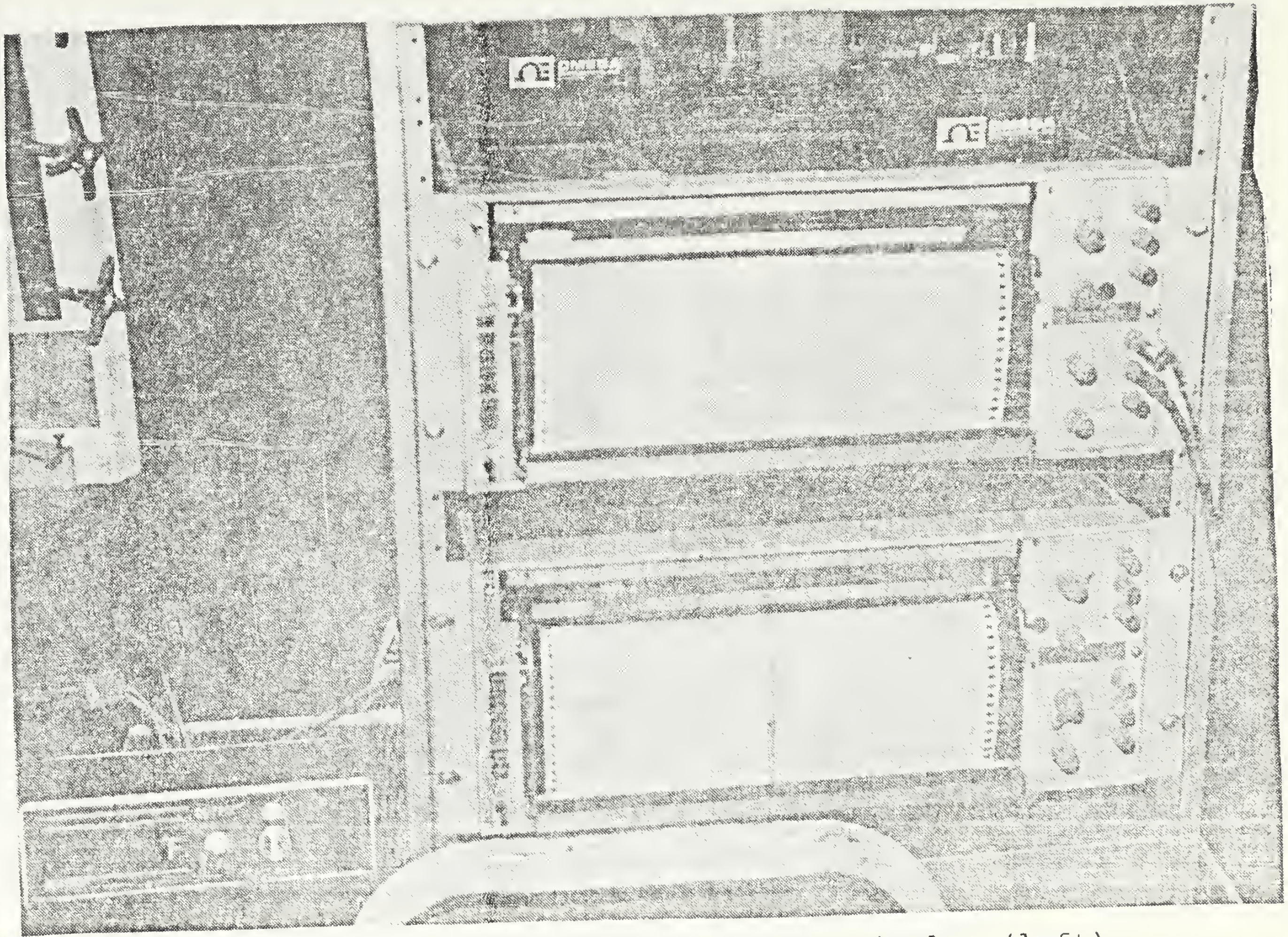

Figure 3. Newport Digital Temperature Display (left) and Strip Chart Recorder (right)

inches of epoxy resin. The 3 by 3 inch and 6 by 6 inch samples required only a small fraction of a can, therefore it was necessary to divide the can by weight fraction and use a portion of the hardener that was consistent with a full mixture as prescribed by the manufacturer. Once the needed amount was removed, the can was closed to reduce any adverse effect that may be caused by extended contact with the environment.

The mixing of CHOCKFAST ORANGE required combining the epoxy resin and the hardener provided in the kit. (Fig. 4) Upon mixing the two components, the mixture is stirred using a heavy duty hand power drill and a type-S mixing blade, provided by Philadelphia Resins Corporation's westcoast distributor. Mixing speed was maintained at a 200 rpm for a period of three minutes. Although a faster mixing speed may ensure a more thorough mixing, it also causes air to be entrapped in the mixture. In all cases, protective gloves and safety glasses were worn when handling the epoxy resin and hardener.

When the mixture was completely mixed, it was poured into the moulds. The mould surfaces were coated with a light coat of CHOCKFAST release agent prior to pouring to prevent adhesion of the chocking material to the mould surfaces. When pouring the mixture, the moulds were held at an angle of approximately ten degrees in order to reduce air entrapment. Although this precaution was taken, the epoxy chocks still showed evidence of air accumulation as could be observed from the air bubbles appearing at the open end of the moulds.

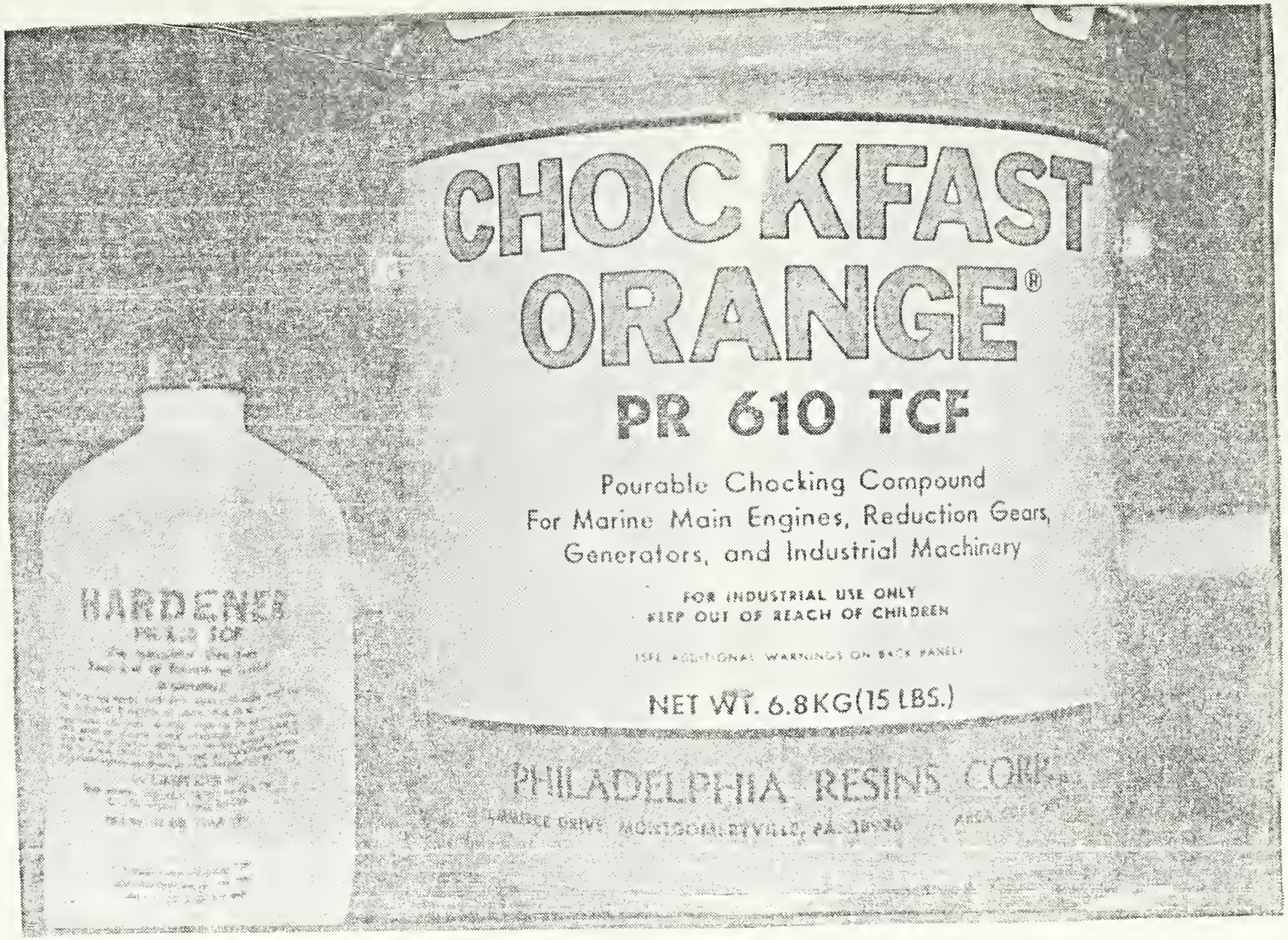

Figure 4. CHOCKFAST ORANGE and Hardener

The effect of entrapped air bubbles on the compressive strength of the chocks is unknown (Fig. 5).

Although the chocks appeared to harden completely in two to three hours after pouring, the chocks were allowed to cure for a period of 24 hours in an ambient temperature of 72-76°F before being removed from the moulds.

D. CUTTING OF EPOXY CHOCKS

Preparation of the compression test specimens required cutting the moulded CHOCKFAST ORANGE epoxy chocks into smaller two inch by two inch blocks. Previous work with epoxy chocks found that using an ordinary band saw generated excess heat along the cutting surface, resulting in possible localized thermal stresses in the epoxy material. Additionally, the epoxy material dulled the blade to the point that replacement of the blade was necessary in a short period of time. Information provided by the manufacturer related that the material is impregnated with fine silica fibers, which causes significant frictional heating and blade wear.

A water cooled, diamond tipped radial saw was used for cutting the samples. The water eliminated the problem of frictional heating and reduced blade vibration due to excess heat. At all times, protective goggles and mask were worn to prevent eye irritation or ingestion of silica dust generated during cutting.

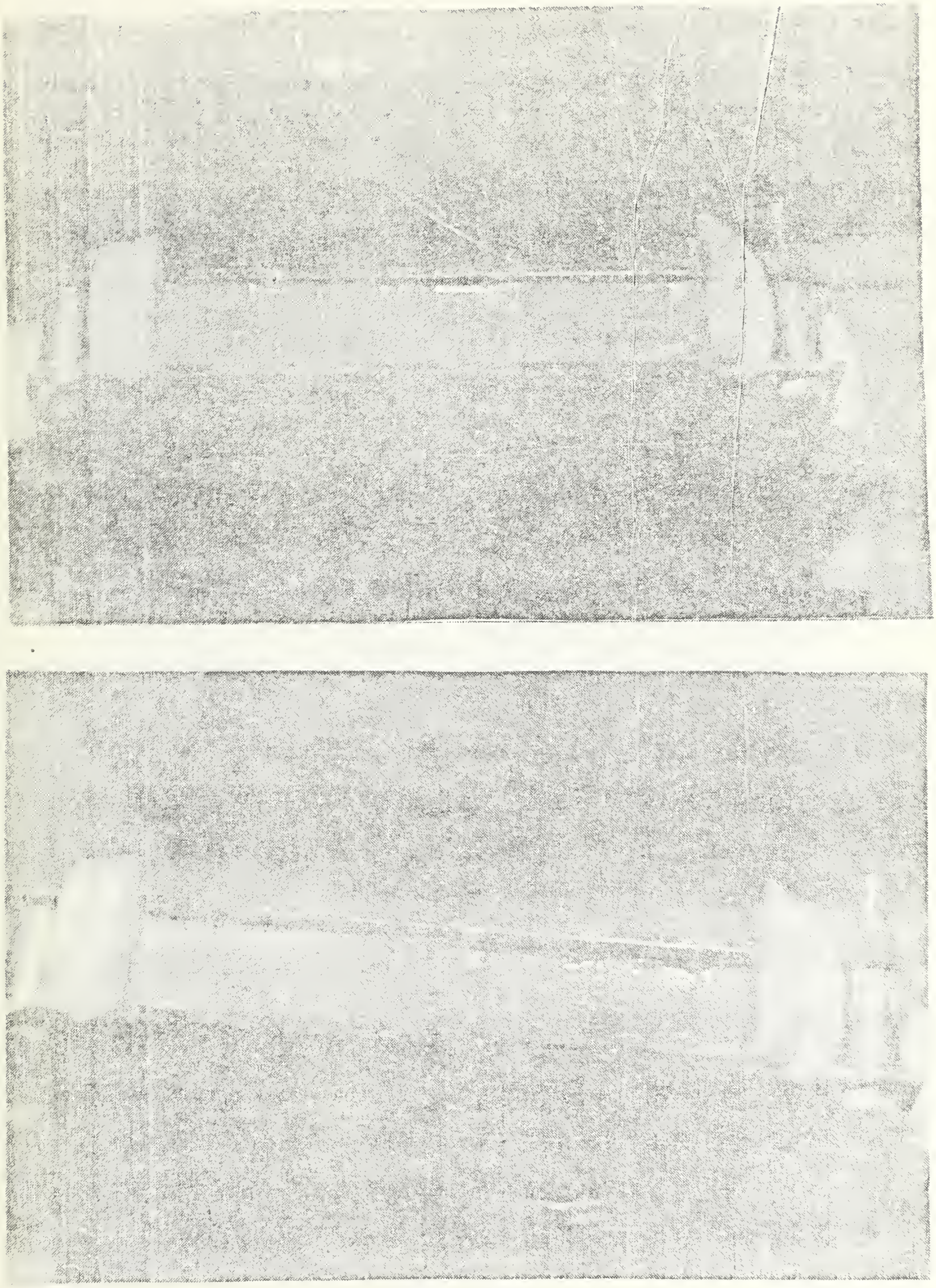

Figure 5. Epoxy Chock as Poured (top)
and After Curing (bottom)

E. COMPRESSIVE TESTING OF EPOXY CHOCKS

The Tinius Olsen 200,000 pound capacity, Super "L" hydraulic testing machine was used for testing all of the 2 inch by 2 inch epoxy specimens (Fig. 6). The machine uses a hydraulic load valve and from a pre-adjusted position will maintain a constant load speed throughout the test. The testing machine did not provide a means of recording compressive deformation of the test samples. A Browne and Sharpe .001 inch graduated, one inch range dial indicator fastened to a brass block provided the necessary deflection data. Due to clearance constraints, it was necessary to use stainless steel shims to support the epoxy test specimen and the dial indicator. (Fig. 7) The dial indicator was zeroed when the crosshead was lowered in position to begin the test run. To ensure the Tinius Olsen test machine was at zero load, corresponding with zero deformation, the load setting dial was placed in the low position. In this position, the maximum allowable load is 4000 pounds, incremented at 5 pounds per division. When the verification of zero load/zero deformation was complete, the test machine was returned to the high scale, corresponding to a maximum load of 200,000 pounds, incremented at 250 pounds per division. All runs were conducted in accordance with ASTM standard D 965, modified to use two inch square test samples. This standard specifies cross head speed will be a constant 1.3±0.3 mm per minute (0.05±0.01 in/min).

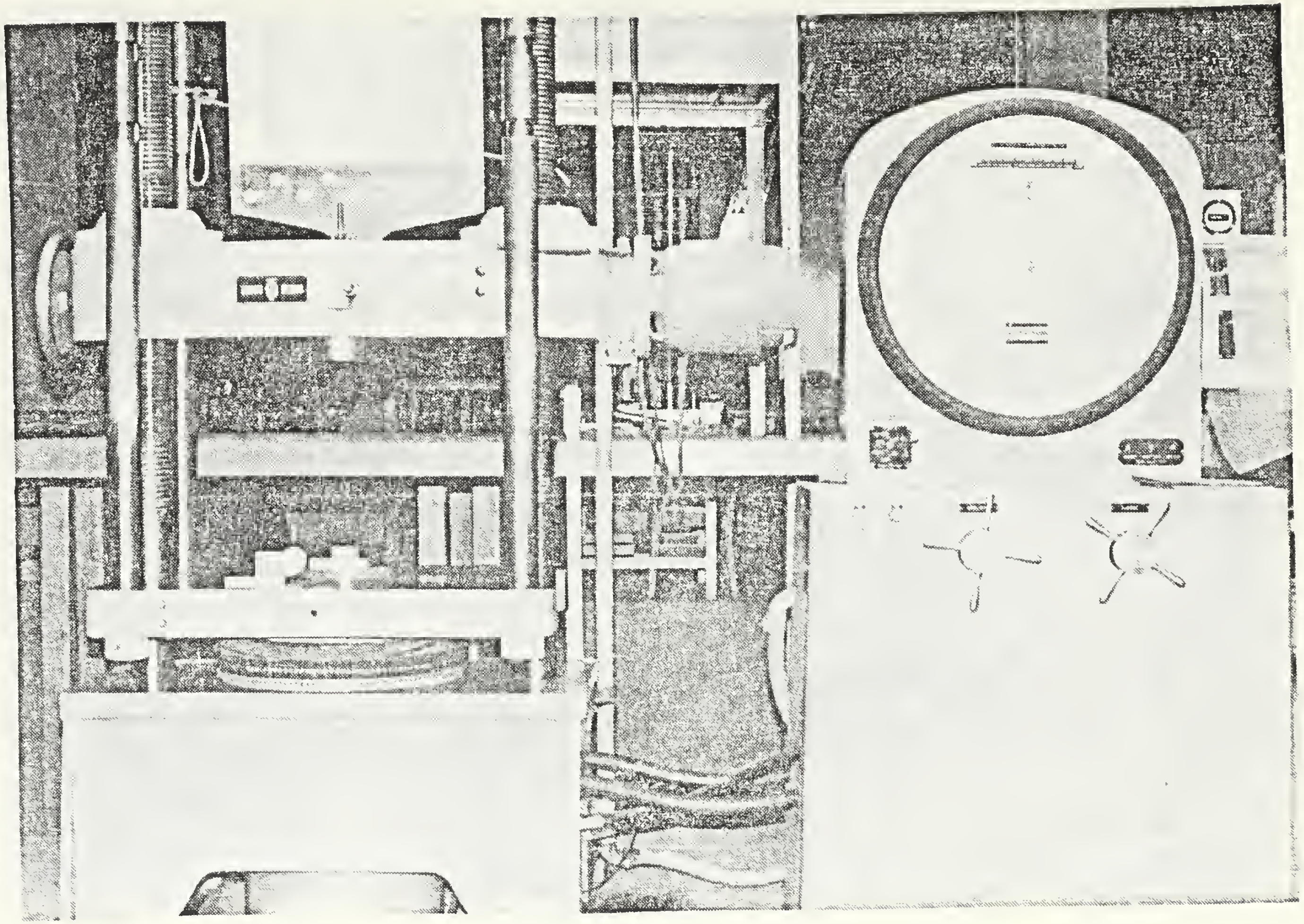

Figure 6. Tinius Olsen Compressive Test Machine

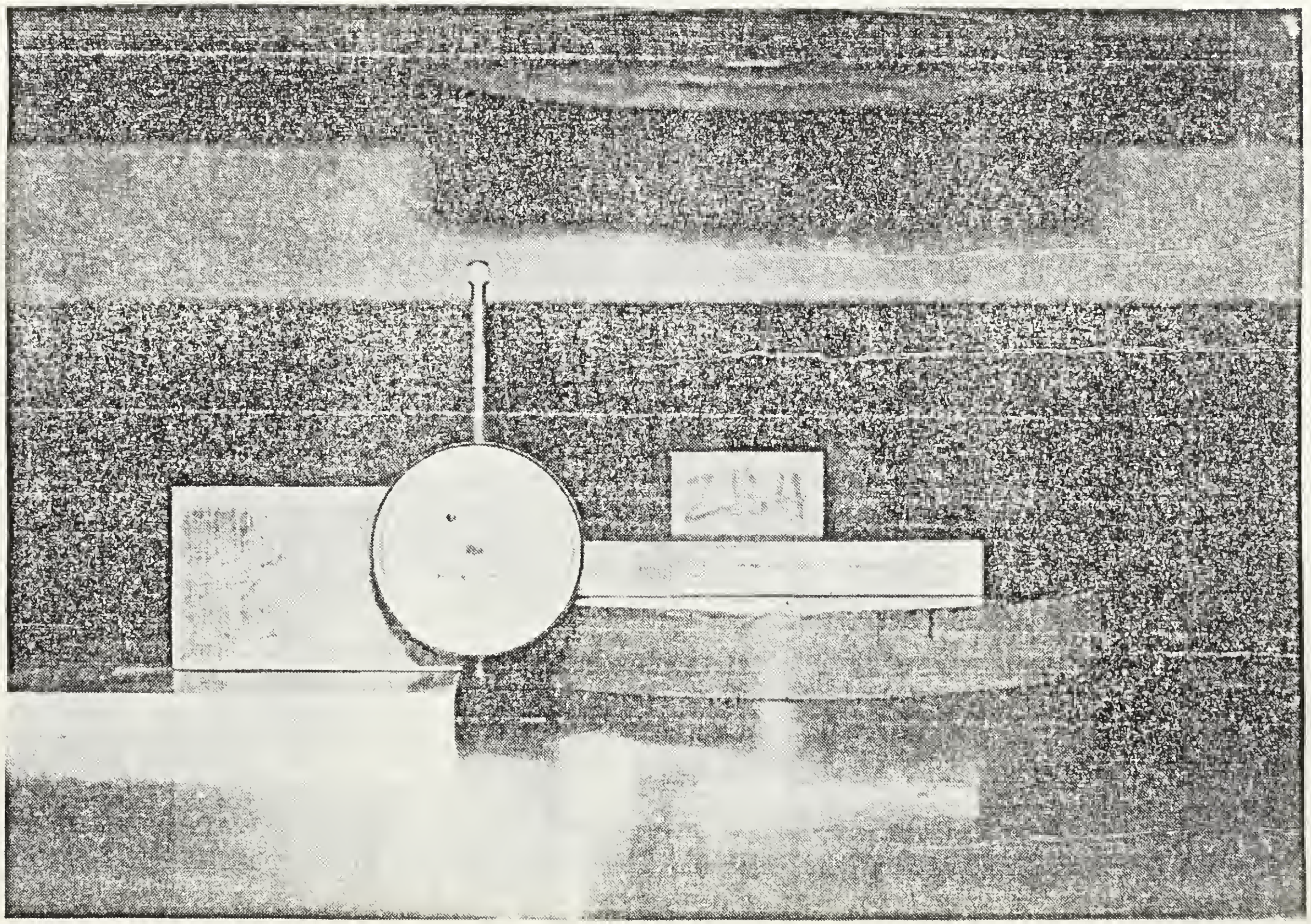

Figure 7. Dial Indicator Arrangement for Measuring Deflection

The compressive testing procedure required two persons to collect the data. One person was responsible for reading the dial indicator and verbally notifying the other person when a specific amount of deformation had occurred. The second person was responsible for controlling the compressive loading and recording the load at the given deformations. A more sophisticated data acquisition system was not available for the compressive test machine used.

A minimum of two test specimens were manufactured from each chock size poured, and with the larger chocks, a greater number of specimens were available. Each test specimen was marked with an alphanumeric code to identify which chock it was cut from, for example "1A1". The first numeral, a 1, 2, or 3, represented the thickness as either 1/2, 1, or 1-1/2 inch. The letter, either an A, B, or C, represented the lateral dimensions 3 by 3, 6 by 6, or 9 by 9 inches. The last numeral determined the pour attempt at which that chock was cut from. An odd number represented the first pour attempt and an even number represented the second pour attempt.

In all cases during the testing, the internal stressing of the test specimen was visibly evident as the compressive load increased into the plastic region. Light colored spots or stars appeared on the sides of the test specimen prior to reaching the ultimate compressive load and failure. Final failure of all samples occurred at the ultimate compressive load. This event was marked by a significant decrease in

load accompanied with a continued increase in plastic deformation. Inspection of the specimens after testing, generally showed cracking along the outer edges of the chock, but no visible cracks generating from the body of the specimen itself. Figures 8 and 9 show the test specimens before and after the compressive testing was conducted.

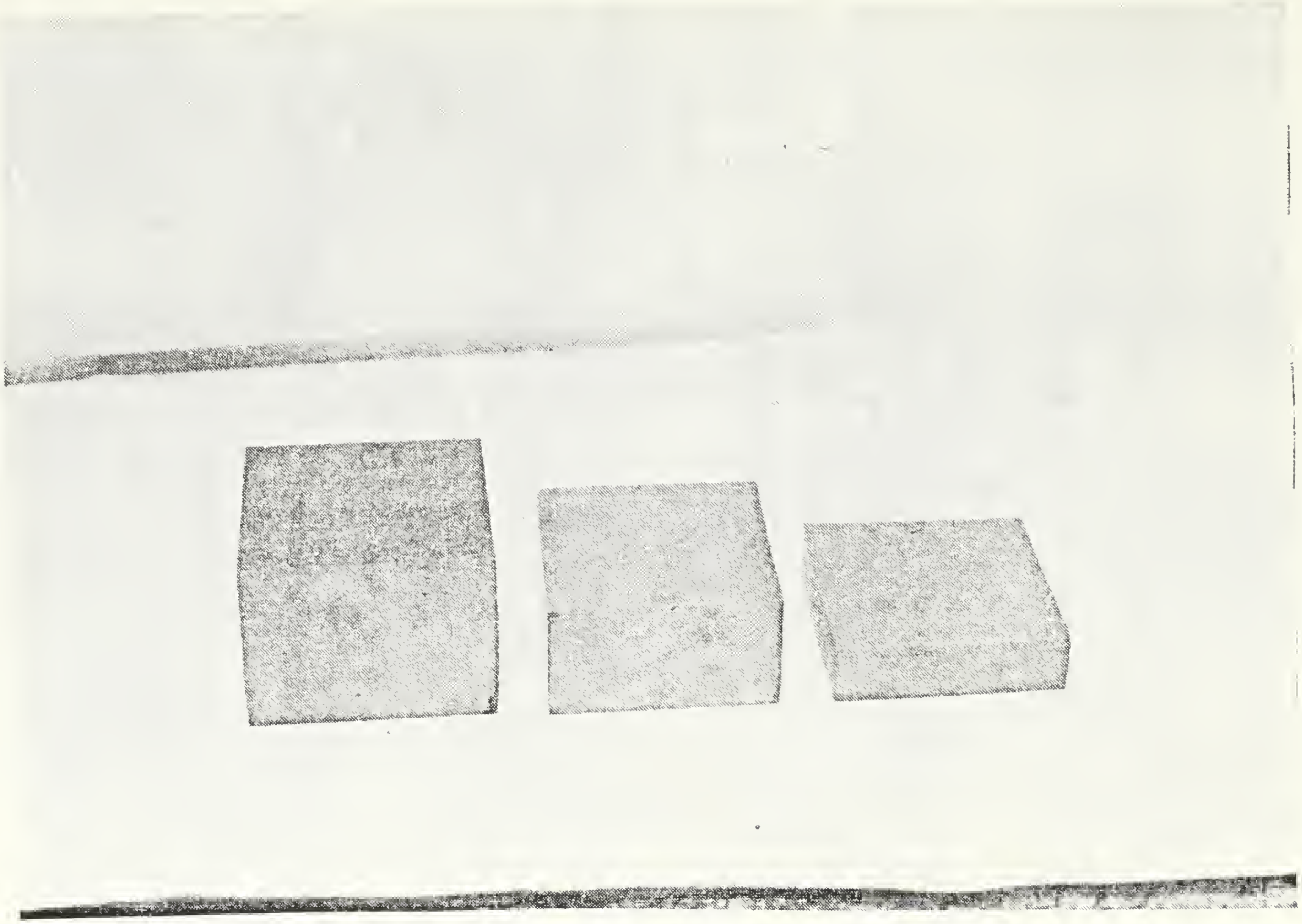

Figure 8. Test Specimens Prior to Compressive Testing

Figure 9. Test Specimens Following Compressive Testing

III. DATA ANALYSIS

A. CALCULATIONS

The thickness of all compressive test specimens were measured in accordance with ASTM D 695-80. Measurements were taken at five locations across the test specimen and the minimum thickness measured was recorded. The measurements were taken using a 0 - 1 inch micrometer (a 1 - 2 inch micrometer was used for the 1-1/2 inch thick samples) accurate to the nearest .001 inch. Measurements of load bearing area were obtained in the same fashion using a 1 - 2 inch micrometer.

The ultimate compressive strength was obtained from the following equation:

Ultimate Compressive Strength = Max Load / Initial Area

Strain measurements recorded as inches per inch were tabulated using the following:

Strain = Deformation / Initial Thickness

Modulus of Elasticity was determined by drawing a tangent to the stress - strain line in the elastic region. Strain, for the purpose of this calculation, was measured from the intersection of the tangent line with the strain axis, in order to eliminate any erroneous data points near the origin. The modulus was then determined by measuring the slope of the tangent line.

Compressive yield strength was determined by the 0.2% offset method.

B. ERROR ANALYSIS

An error analysis was conducted for the stress and strain calculations and is provided in Appendix A. The uncertainties used are provided below:

Compressive load	± 125 Lbf
Dimension measurements	± 0.0005 in.
Deformation	± 0.0015 in.

IV. RESULTS

A. TEMPERATURE RESULTS

With all three lateral dimension sizes, the maximum temperature reached during the curing process increased with increased mould thickness. The most dramatic and probably the most representative of what can be expected were the 6 x 6 inch test samples. The 1/2 inch thick sample showed very little temperature rise, whereas the 1 inch and 1-1/2 inch thick samples resulted in a very large exothermic reaction. With both 1 inch and 1-1/2 inch thicknesses the shape of the time - temperature curve was similar. The slope of the curve remained fairly constant in the early stages of the reaction, until the temperature reached approximately 120^{o}F, when the slope increased exponentially until the maximum.

The maximum temperature was marked by the solidification of the chocking compound. In the case of all of the samples, the compound became increasingly viscous with time. However, with the 1/2 inch thick samples, there was no apparent transition from the liquid to the solid state. In contrast, in the samples that generated steep temperature gradients, the compounds remained "tacky" to the touch until the maximum temperature was reached. At that point, a visible transition could be seen generating from the center of the exposed surface and spreading outward in all directions. The transition

was characterized by a darkening of the chocking compound and a change in the appearance from a tacky texture to a hard crust like texture. Additionally, the amount of air or gas bubbles present on the surface increased drastically. Results of the 6 by 6 inch samples are shown below in Table III. The temperature vs. time graphs for the 6 by 6 inch series are shown in Figures 10, 11, and 12.

Table III.

Exotherm Results of 6 by 6 Inch Samples

THICKNESS (IN)	VOLUME (IN^3)	Tmax (^{O}F)	TIME (MIN)
1/2	18.0	103.2	139.0
1/2	18.0	102.4	131.0
1	36.0	273.4	66.5
1	36.0	250.6	62.5
1-1/2	54.0	331.5	55.0
1-1/2	54.0	327.3	51.5

The results of the 3 by 3 inch and 9 by 9 inch samples were markedly different from those obtained with the 6 by 6 inch samples. The 3 by 3 inch samples showed no significant exothermic reaction for either the 1/2 inch or 1 inch samples. In the case of the 3 by 3 by 1-1/2 inch sample, as was seen with the 6 by 6 by 1 inch and the 6 by 6 by 1-1/2 inch samples, the temperature rose steadily in the early stages

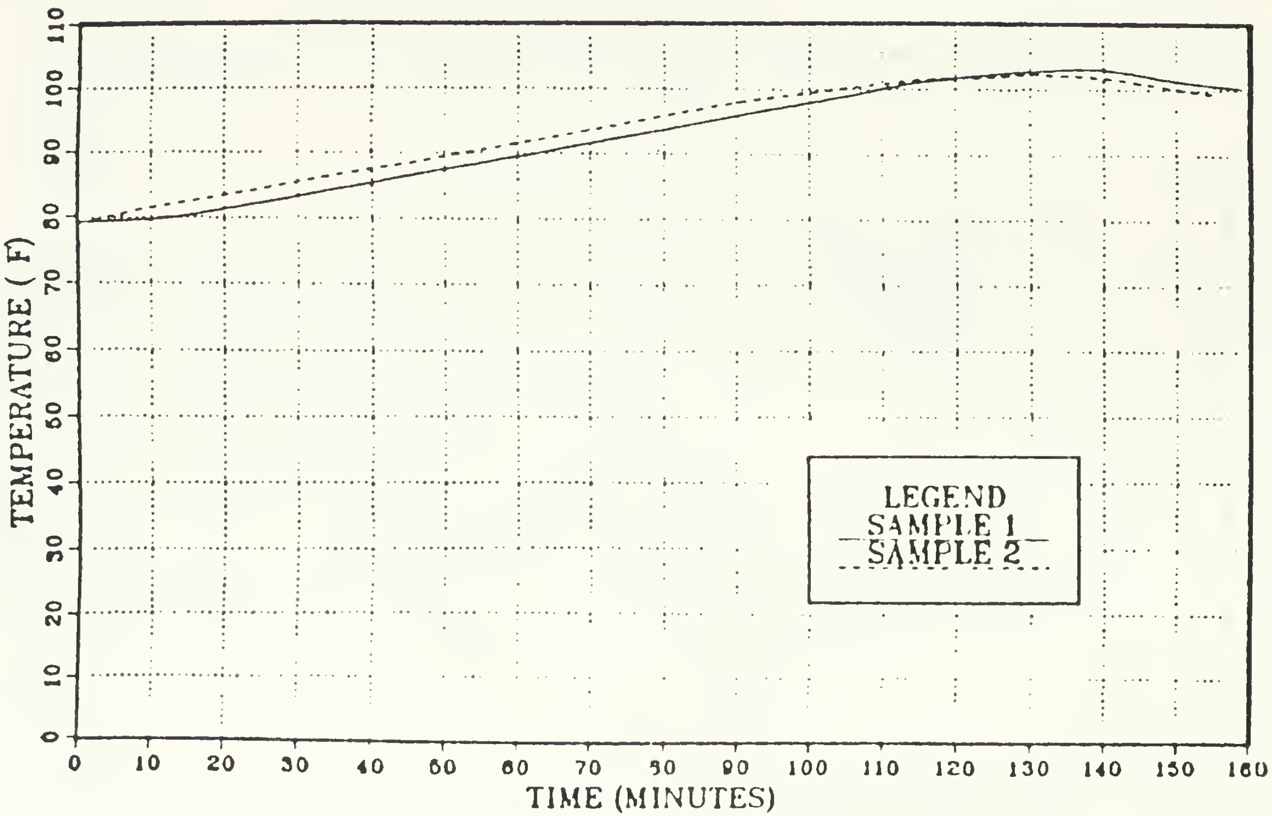

Figure 10. Temperature vs. Time Graph of 6x6x1/2 Inch Samples

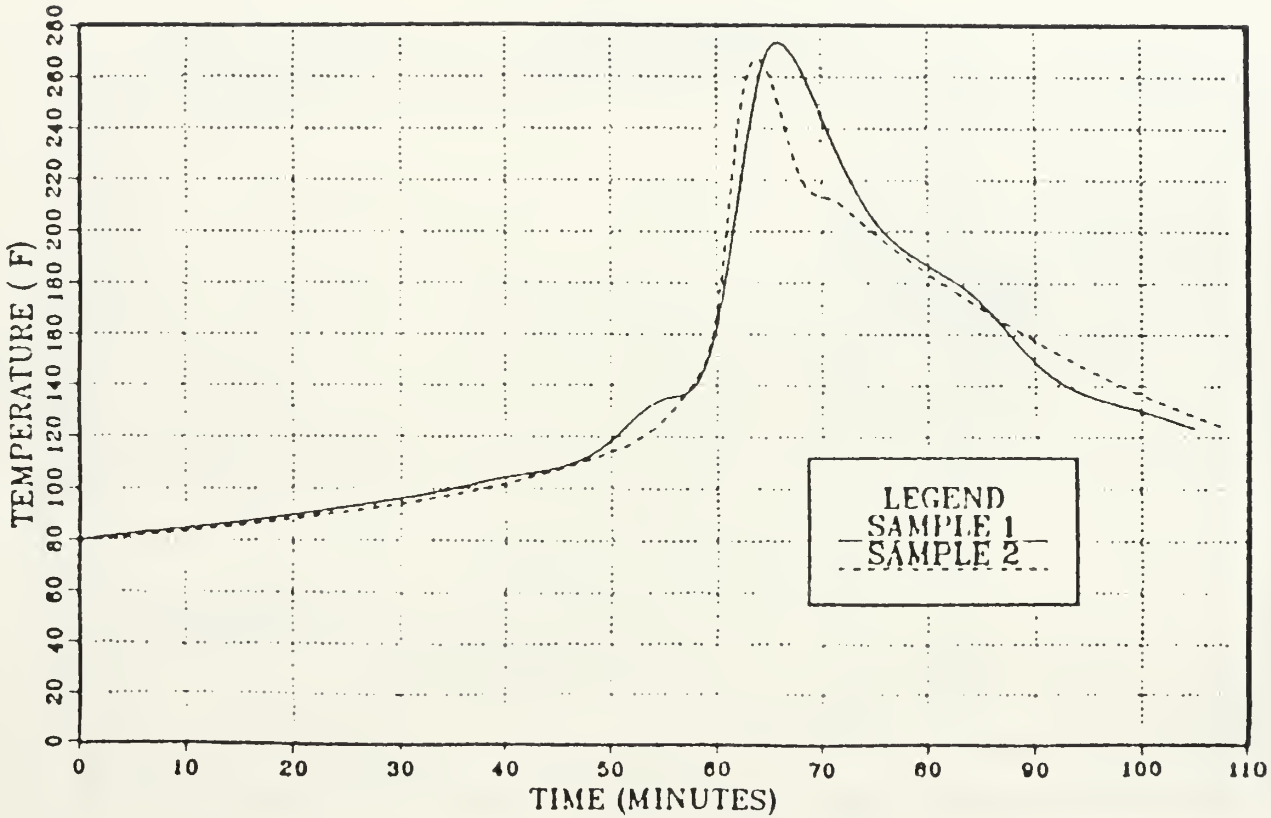

Figure 11. Temperature vs. Time Graph of 6x6x1 Inch Samples

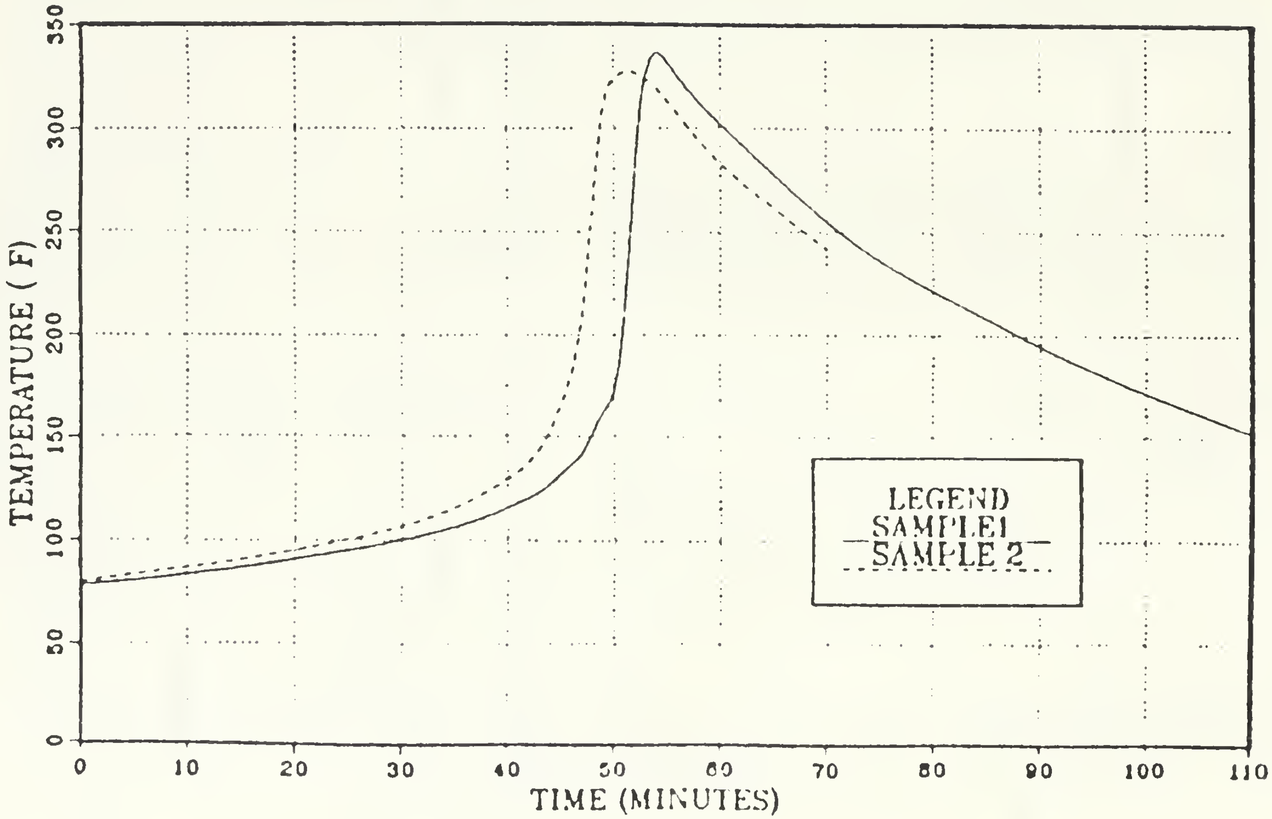

Figure 12. Temperature vs. Time Graph of 6x6x1-1/2 Inch Samples

of the curing process and experienced the exponential rise as was mentioned. Results of the 3 by 3 inch samples are shown below in Table IV. The temperature vs. time graphs are shown in Figures 13, 14, and 15.

Table IV.

Exotherm Results of 3 by 3 Inch Samples

THICKNESS (IN)	VOLUME (IN^3)	Tmax (^{O}F)	TIME (MIN)
1/2	4.5	84.5	139.0
1/2	4.5	85.7	137.0
1	9.0	108.6	99.0
1	9.0	98.0	107.0
1-1/2	13.5	271.2	66.5
1-1/2	13.5	268.7	67.5

The results of the 9 by 9 inch test samples are shown below in Table V., with the temperature vs. time graphs provided in Figures 16, 17, and 18.

Table V.

Exotherm Results of 9 by 9 Inch Samples

THICKNESS (IN)	VOLUME (IN^3)	Tmax (^{O}F)	TIME (MIN)
1/2	40.5	95.9	140.0
1	81.0	185.0	59.0
1-1/2	121.5	324.3	79.0

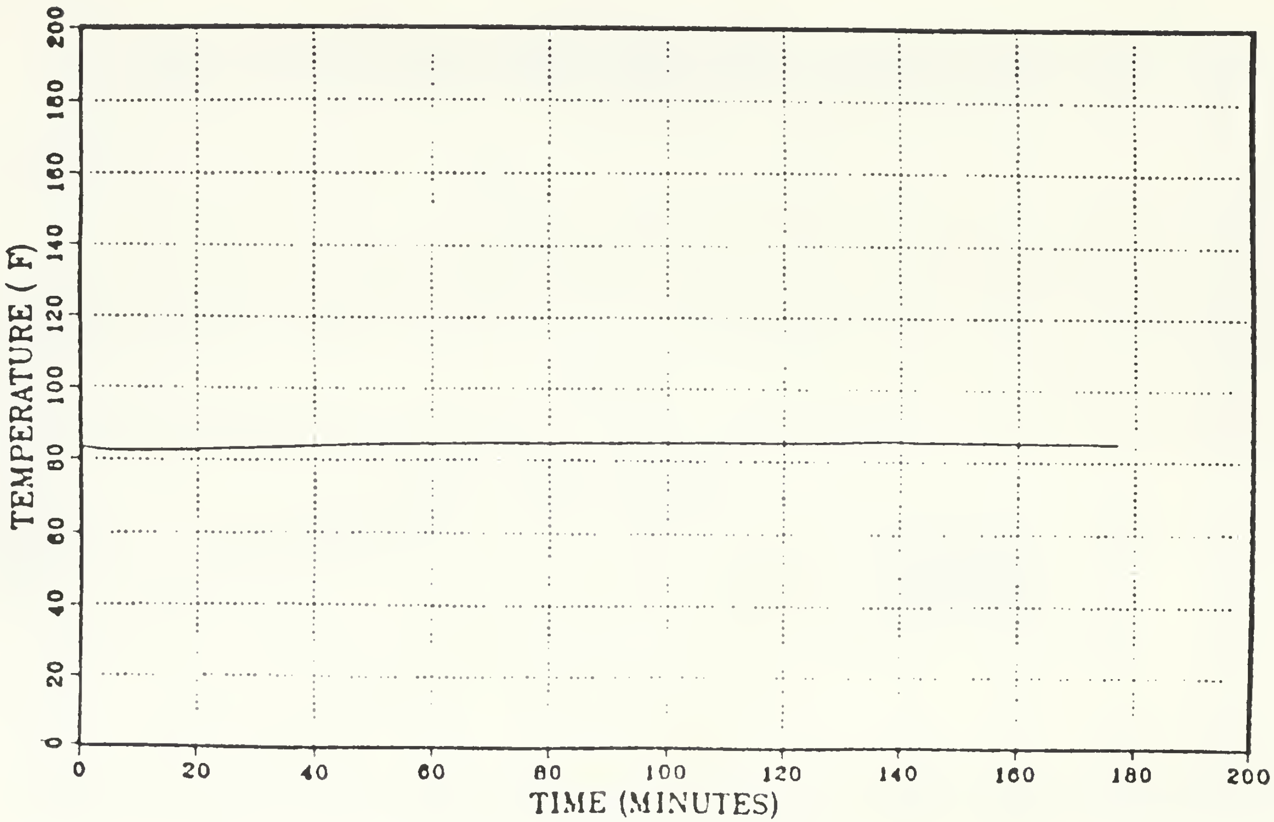

Figure 13. Temperature vs. Time Graph of 3x3x1/2 Inch Samples

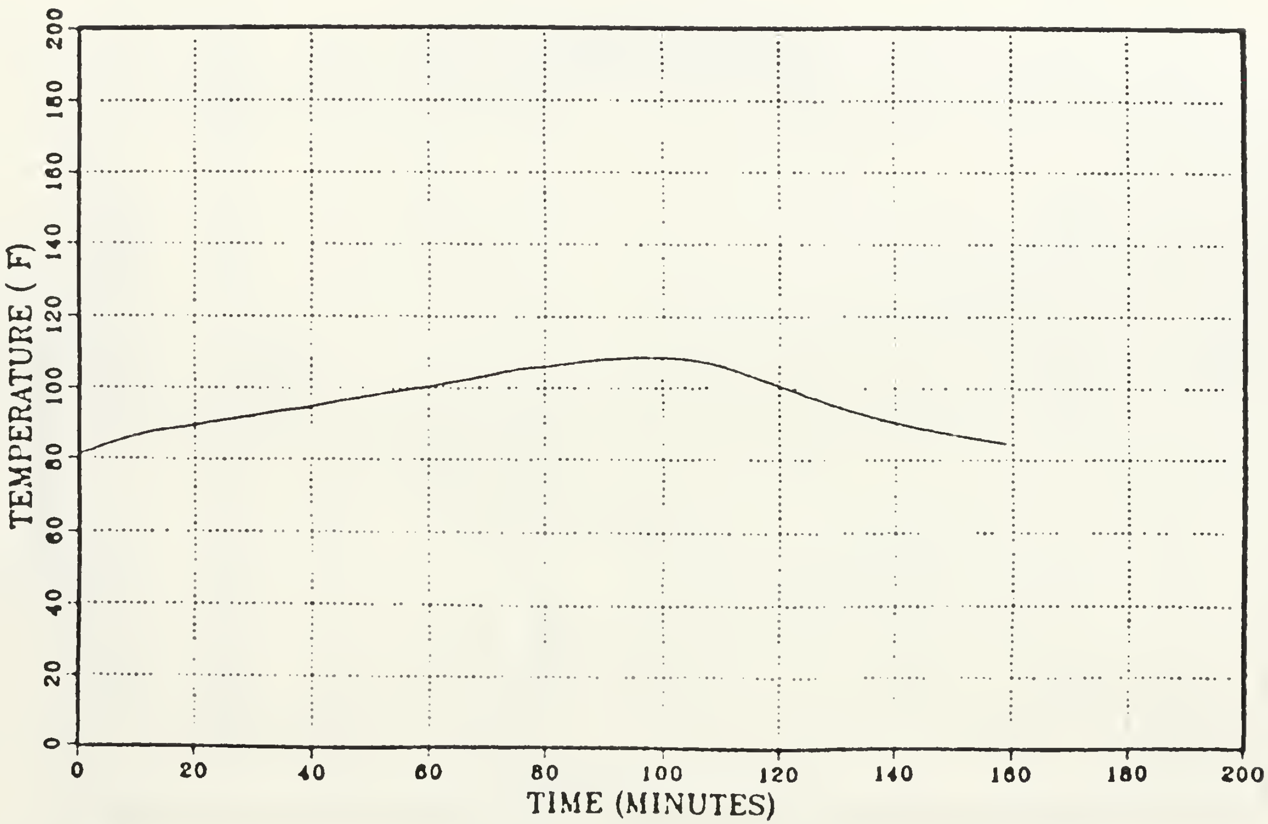

Figure 14. Temperature vs. Time Graph of 3x3x1 Inch Samples

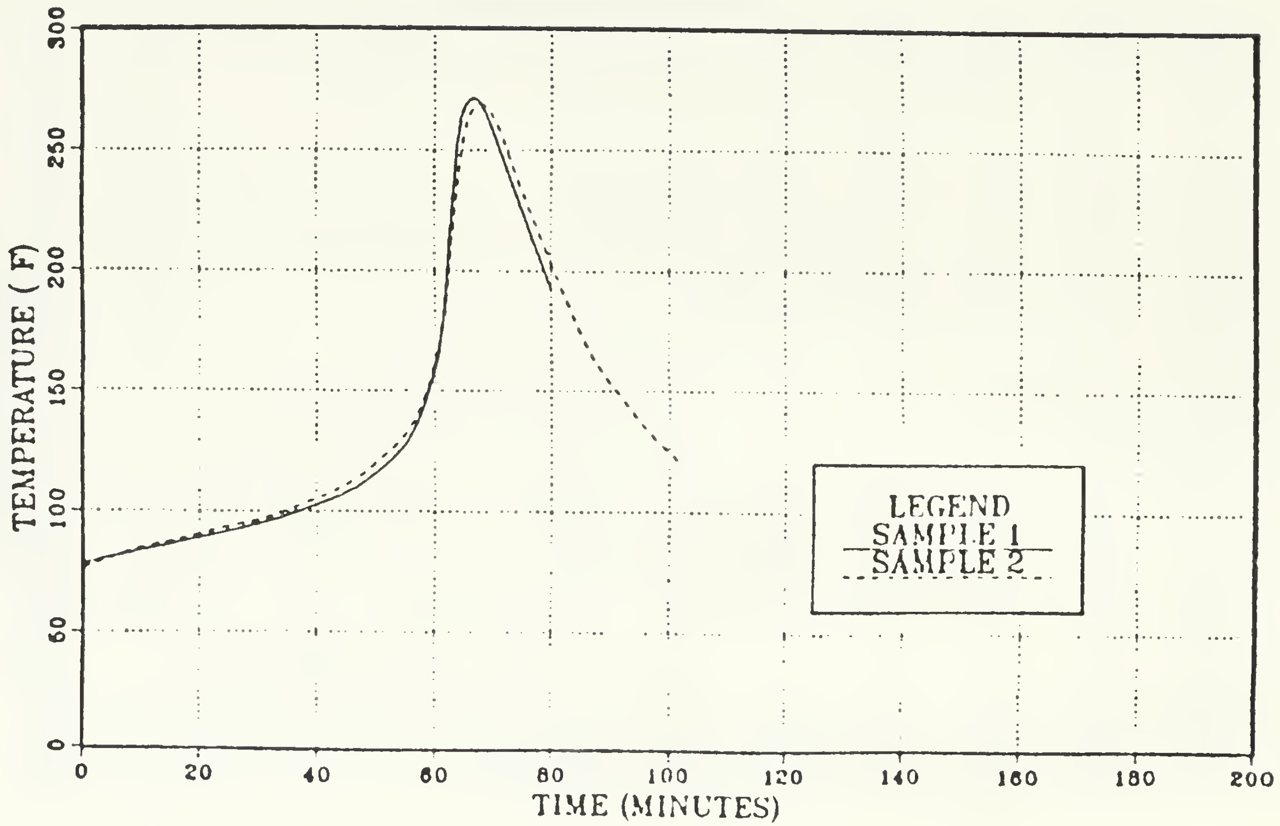

Figure 15. Temperature vs. Time Graph of 3x3x1-1/2 Inch Samples

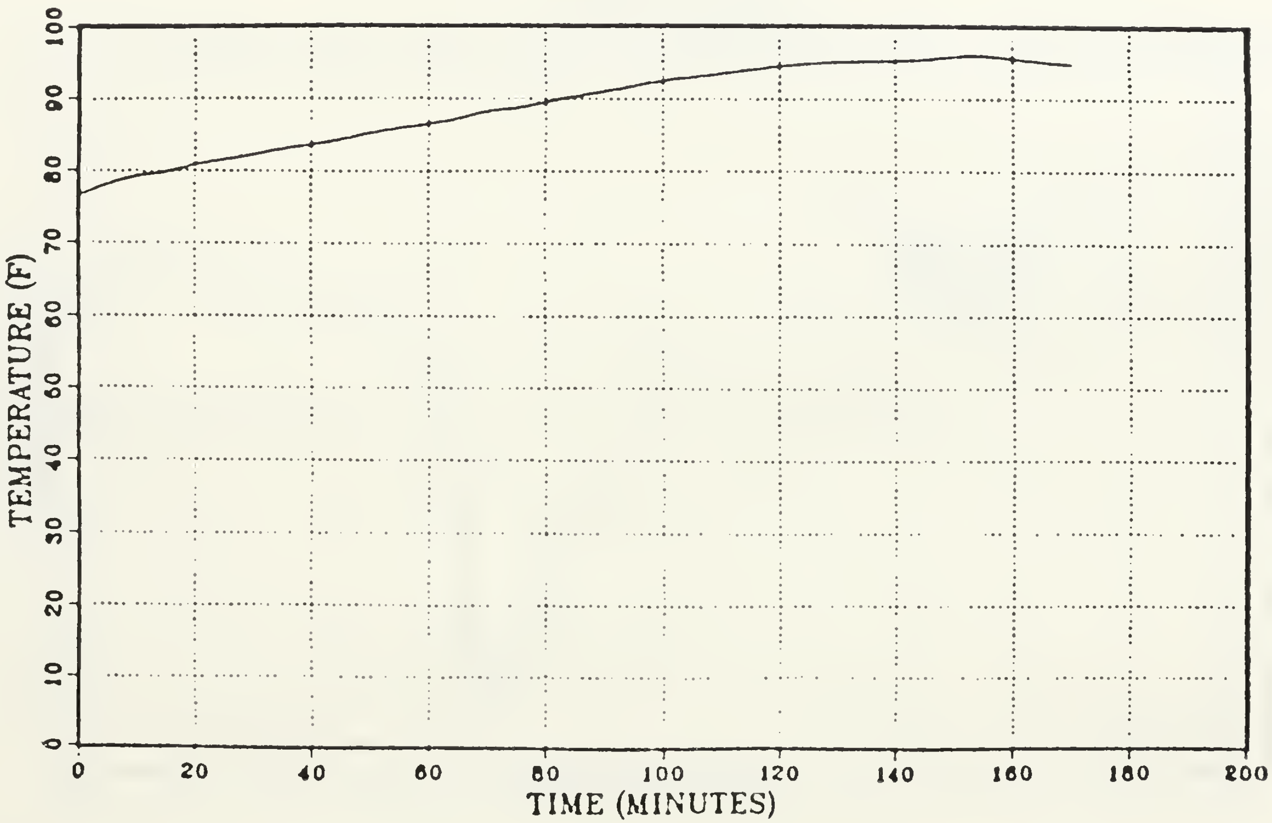

Figure 16. Temperature vs. Time Graph of 9x9x1/2 Inch Samples

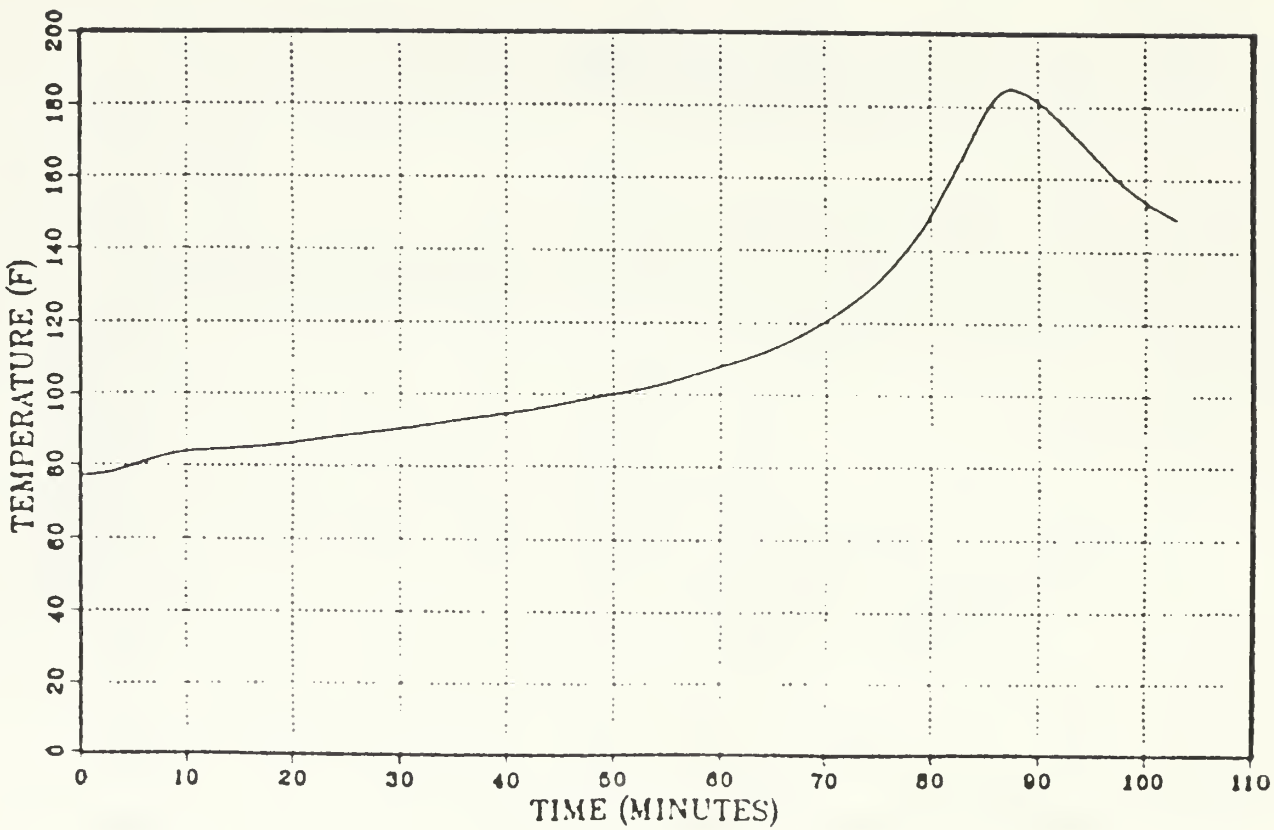

Figure 17. Temperature vs. Time Graph of 9x9x1 Inch Samples

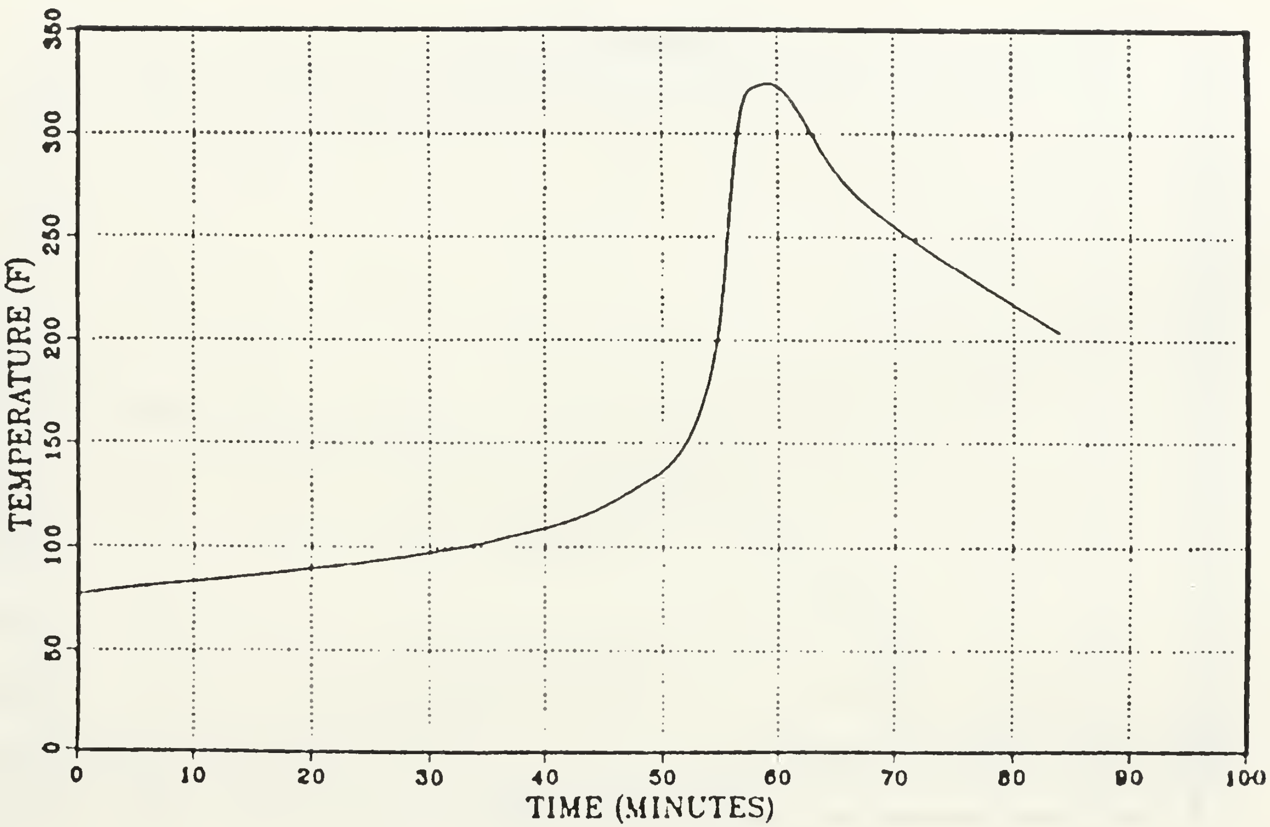

Figure 18. Temperature vs. Time Graph of 9x9x1-1/2 Inch Samples

For the 3 by 3 inch and 9 by 9 inch samples, the results of the 1/2 inch and 1 inch samples were lower than expected and caused some concern in providing an explanation of the cause. The following explanation is provided as a possible reason.

In general, epoxy resins are poor thermal conductors. The curing process involves the polymerization of the chocking compound, resulting in the transformation of the epoxy from a liquid to a solid. With this transformation, energy is released in the form of heat. The outside layers of the chocking compound adjacent to the mould walls are cooled by the surroundings, thus forcing the center of the mould to rise to a higher temperature than the outer surfaces in order to force the heat outward. In the case of the 3 by 3 by 1 inch and the 9 by 9 by 1 inch samples, which were poured in castings with larger lateral dimensions than the samples themselves, the plates acted in effect as fin surfaces to more efficiently dissipate the heat being generated. Thus keeping the temperature rise in check. For the 6 by 6 by 1 inch sample, where the epoxy completely filled the casting, the resultant temperature rise was much greater.

To support this hypothesis, two samples were poured from the same mixture, one in the small mould casting and the second in the larger casting. Both samples measured 6 by 6 by 1 inch with the only variation being the size of the casting. The sample cast in the smaller mould reached a

maximum temperature of 243.7°F, while the sample in the larger casting reached a maximum temperature of 112.0°F.

B. RESULTS OF COMPRESSIVE TESTS

The testing of all test specimens provided a well defined elastic and plastic region. The 1/2 inch specimens were characterized by a very short plastic region prior to failure. Inspection of the specimens following testing showed no visible signs of failure. Failure was determined by a substantial decrease in compressive load accompanied by an increase in deformation. The tabulated results of the compressive tests of the 1/2 inch samples are provided in Table VI. and the stress - strain curves are provided in Figures 19, 20, and 21.

The 1 inch thick test specimens, as reported earlier, had the greatest variation in exotherm. Corresponding to that was a sizeable increase in the ultimate compressive strength. The test specimens were characterized by a substantial increase in plastic deformation prior to failure with the increased ultimate strengths. Visual inspection of the failed specimens showed cracks forming at the edges of the specimen, extending into the body. Tabular results are shown in Table VII. and the corresponding stress - strain curves are shown in Figures 22, 23, and 24.

Although the exothermic reaction achieved by 1-1/2 inch thick samples increased over the 1 inch thick samples, the

Table VI.

Ultimate Strength Table for 1/2 Inch Test Specimens

SPECIMEN	AREA (IN^2)	THICKNESS (IN)	MAXIMUM LOAD (LBF)	ULTIMATE STRENGTH (PSI)	STRAIN
1A1	4.250	0.502	69,100	16,244	.0837
1A2	3.996	0.511	68,750	17,054	.0548
1A3	3.899	0.522	63,900	16,390	.0958
	MEAN ULTIMATE STRENGTH		16,563		
1B1	3.847	0.536	65,300	16,972	.0933
1B2	3.942	0.552	66,900	16,970	.0996
	MEAN ULTIMATE STRENGTH		16,971		
1C1	3.967	0.534	68,600	17,291	.0936
1C2	3.987	0.536	69,250	17.772	.0746
	MEAN ULTIMATE STRENGTH		17,532		

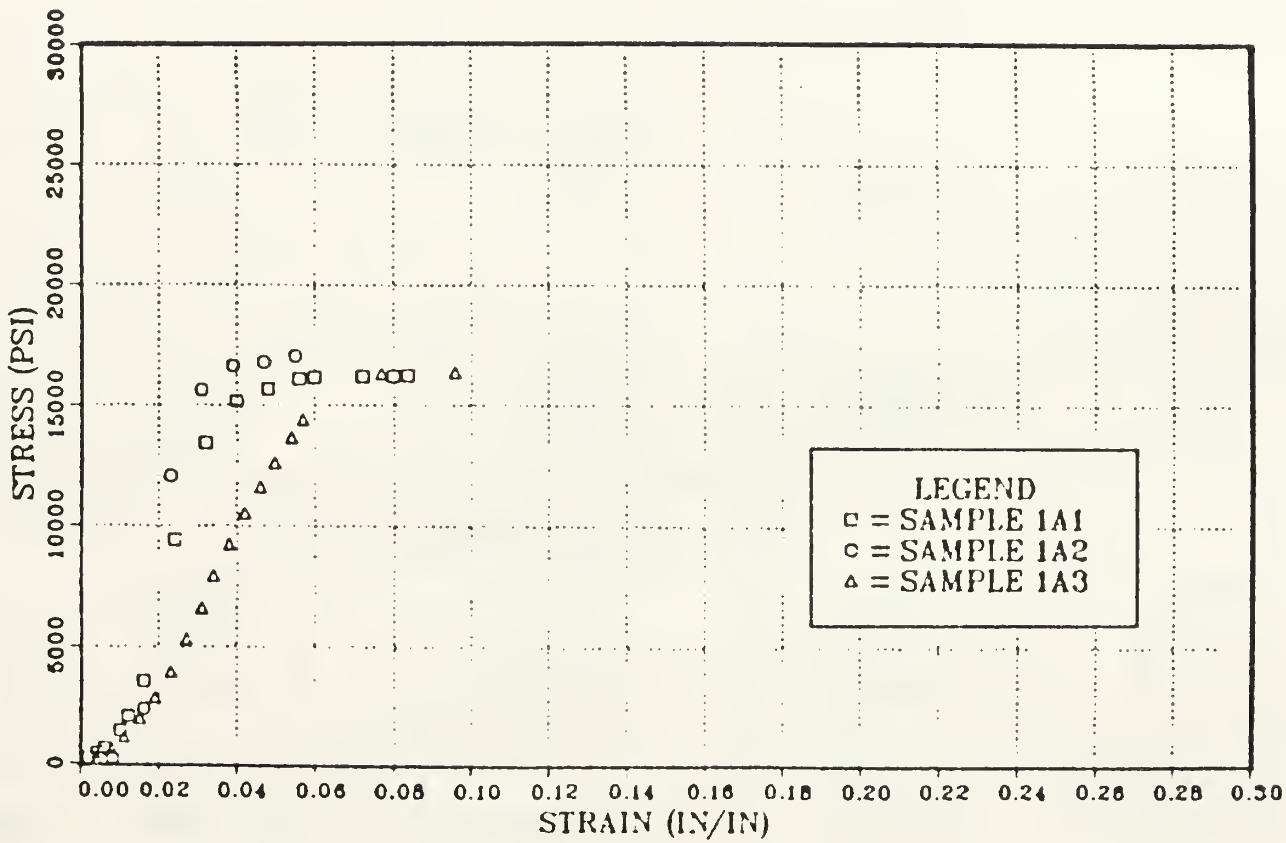

Figure 19. Stress-Strain Curve, 3x3x1/2 Inch Samples

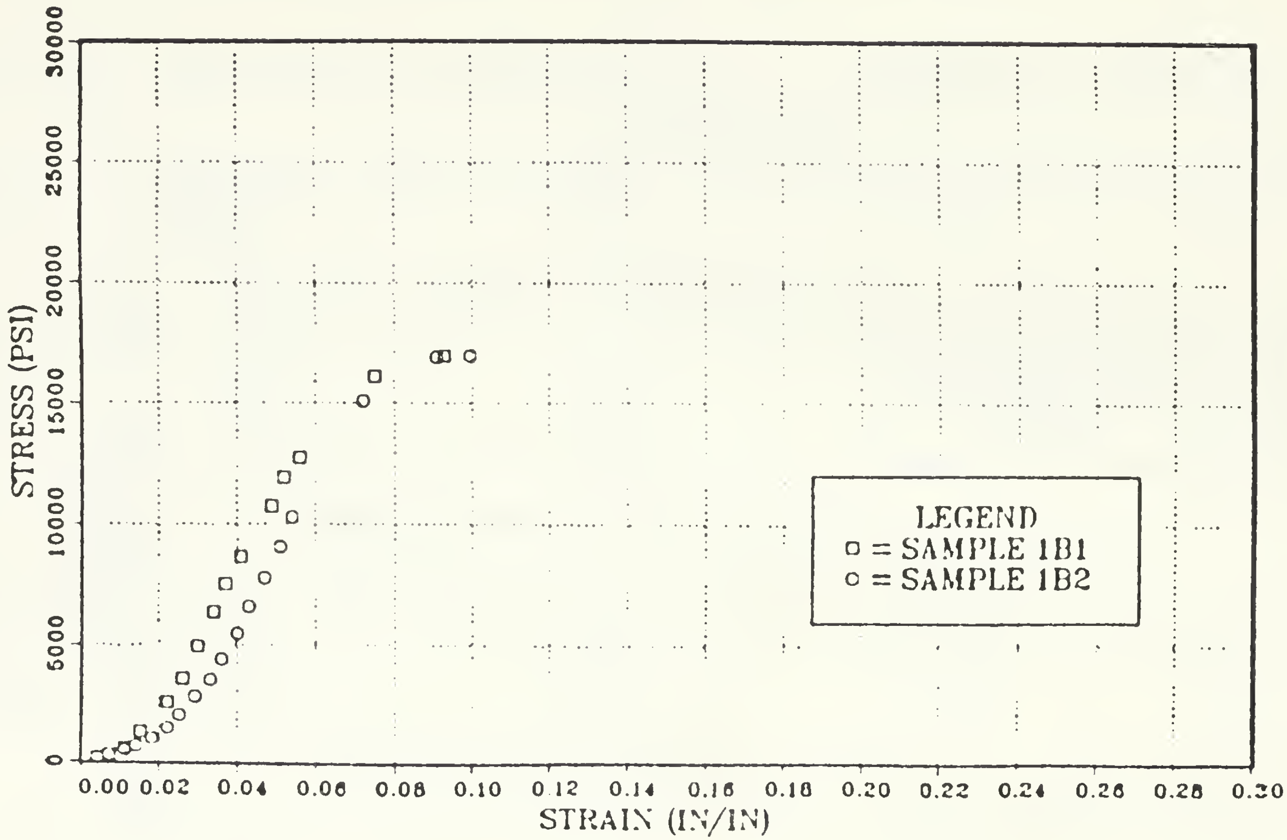

Figure 20. Stress-Strain Curve, 6x6x1/2 Inch Samples

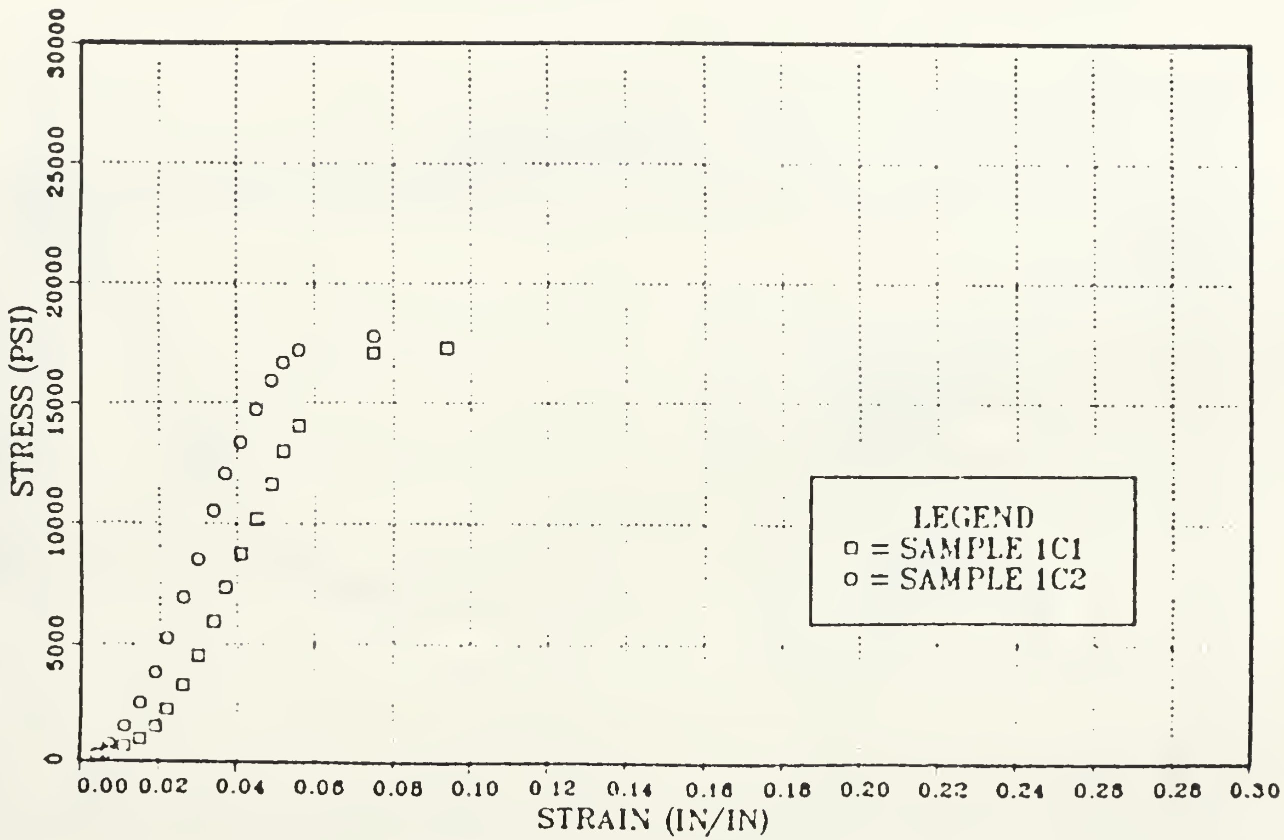

Figure 21. Stress-Strain Curve, 9x9x1/2 Inch Samples

Table VII.

Ultimate Strength Table for 1 Inch Test Specimens

SPECIMEN	AREA (IN^2)	THICKNESS (IN)	MAXIMUM LOAD (LBF)	ULTIMATE STRENGTH (PSI)	STRAIN
2A1	3.959	1.007	60,500	15,281	.0596
2A2	3.822	1.011	60,000	15,699	.0593
	MEAN ULTIMATE STRENGTH	15,490			
2B1	3.960	0.998	99,750	25,189	.2204
2B2	3.881	1.009	98,750	25,446	.3271
2B3	3.910	0.998	98,250	25,129	.2305
	MEAN ULTIMATE STRENGTH	25,255			
2C1	3.958	1.002	82,250	20.783	.2345
2C2	3.959	1.001	87,750	22,164	.2697
2C4	3.952	1.012	85,900	21,691	.2470
	MEAN ULTIMATE STRENGTH	21,546			
LM *	3.952	0.997	60,900	15,411	.0702

* Specimen LM was cut from the 6x6x1 inch chock cast in the large mould.

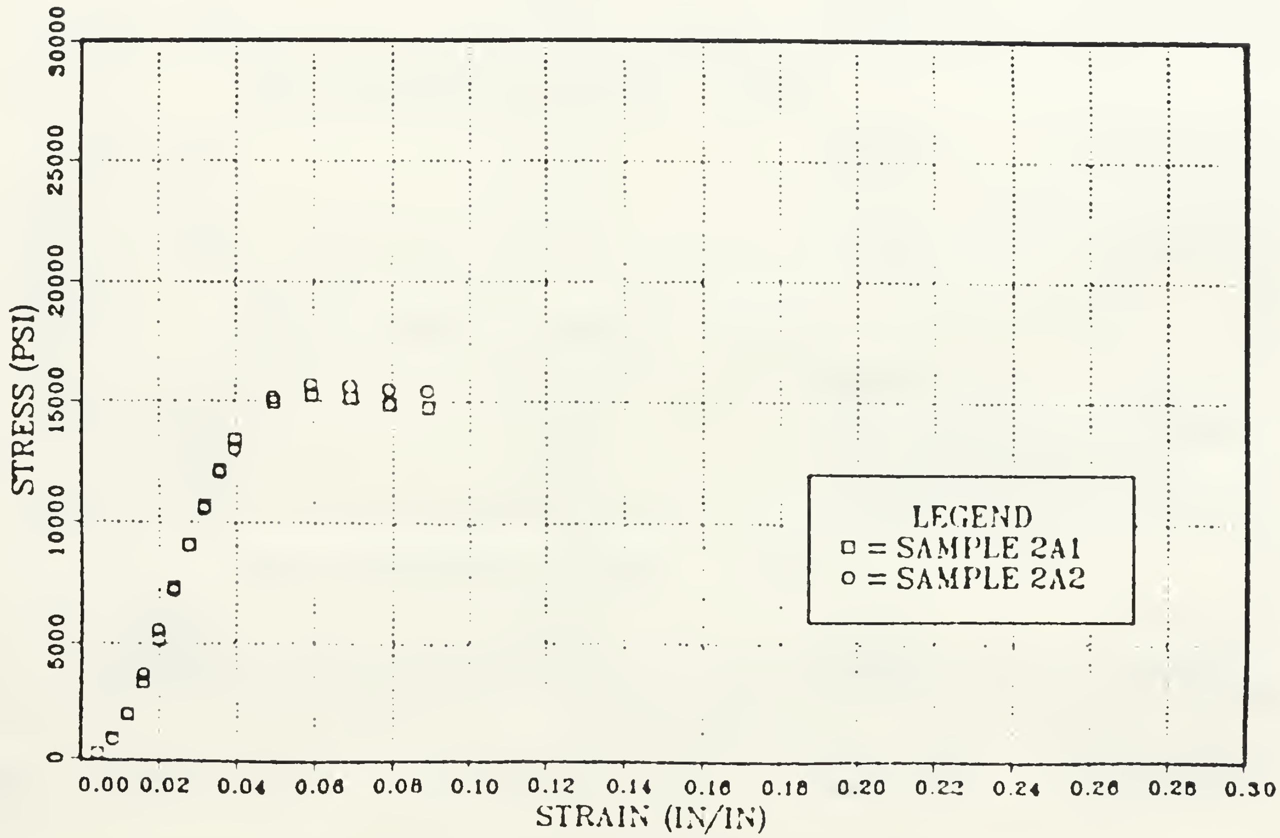

Figure 22. Stress-Strain Curve, 3x3x1 Inch Samples

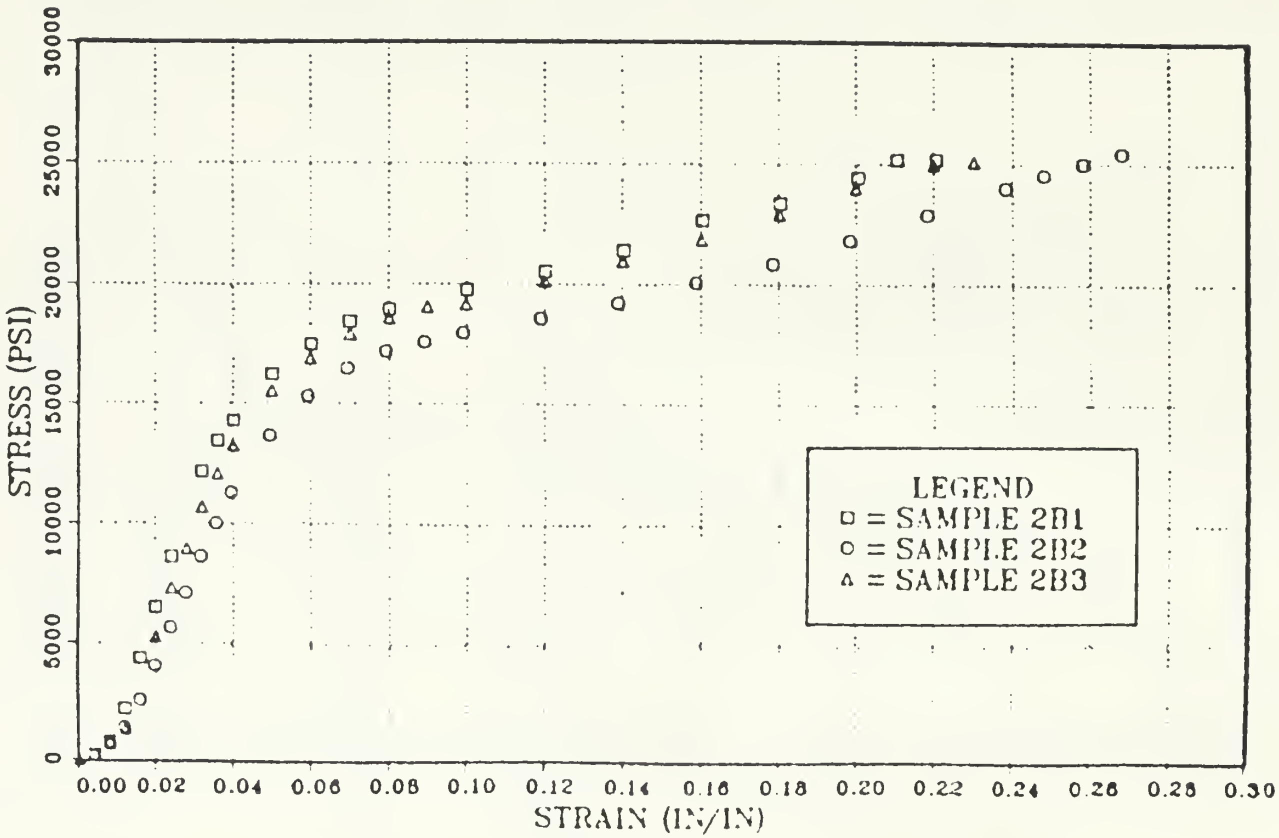

Figure 23. Stress-Strain Curve, 6x6x1 Inch Samples

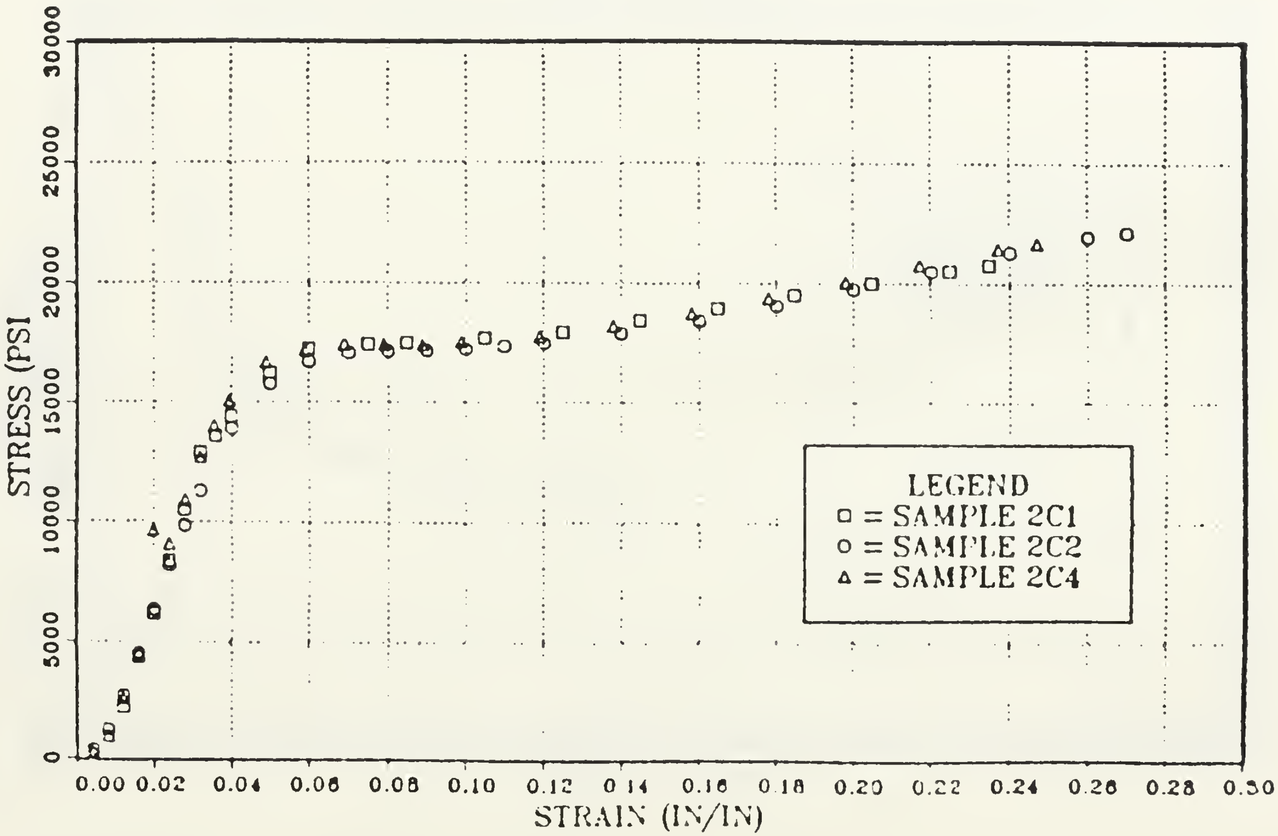

Figure 24. Stress-Strain Curve, 9x9x1 Inch Samples

ultimate strength decreased slightly. Speculation into the cause of the decreased strength is the presence of entrapped air in the samples. Because of the higher exotherms, the reaction rate increases as well. This means that curing occurs much faster and air or gas bubbles generated in the center of the sample do not have sufficient time to escape, thus reducing the strength of the chock.

An analysis of the stress-strain curves for the 1-1/2 inch specimens shows a large plastic region before failure occurs, as in the 1 inch thick samples. Visual inspection of the failed specimens again showed cracks generating from the edges of the specimen, however in two incidences the test specimens had areas separated totally from the specimen, indicating failure may have initiated from a void within the body of the specimen. Tabular results are provided in Table VIII. and the stress-strain curves are shown in Figures 25, 26, and 27.

C. MODULUS OF ELASTICITY AND COMPRESSIVE YIELD STRENGTH

The results for modulus of elasticity and compressive yield strength are shown in Table IX.

The modulus of elasticity was determined from the plot of stress-strain in the ealstic region for each specimen. A mean value was tabulated for each specific chock size. No clear conclusion on the effects of exotherm can be drawn from the obtained results. A slight increase in elastic

Table VIII.

Ultimate Strength Table for 1-1/2 Inch Thick Test Specimens

SPECIMEN	AREA (IN^2)	THICKNESS (IN)	MAXIMUM LOAD (LBF)	ULTIMATE STRENGTH (PSI)	STRAIN
3A1	3.548	1.499	79,100	22,294	.2502
3A2	3.758	1.508	80,300	21,369	.2056
		MEAN ULTIMATE STRENGTH	21,832		
3B1	4.016	1.480	86,500	21,539	.1824
3B2	4.046	1.488	90,250	22,305	.1546
3B3	3.892	1.478	82,800	21,272	.1759
		MEAN ULTIMATE STRENGTH	21,705		
3C1	4.024	1.491	89,400	22,217	.1945
3C2	3.988	1.489	92,500	23,196	.2015
3C3	3.910	1.479	89,650	22,927	.2164
		MEAN ULTIMATE STRENGTH	22,780		

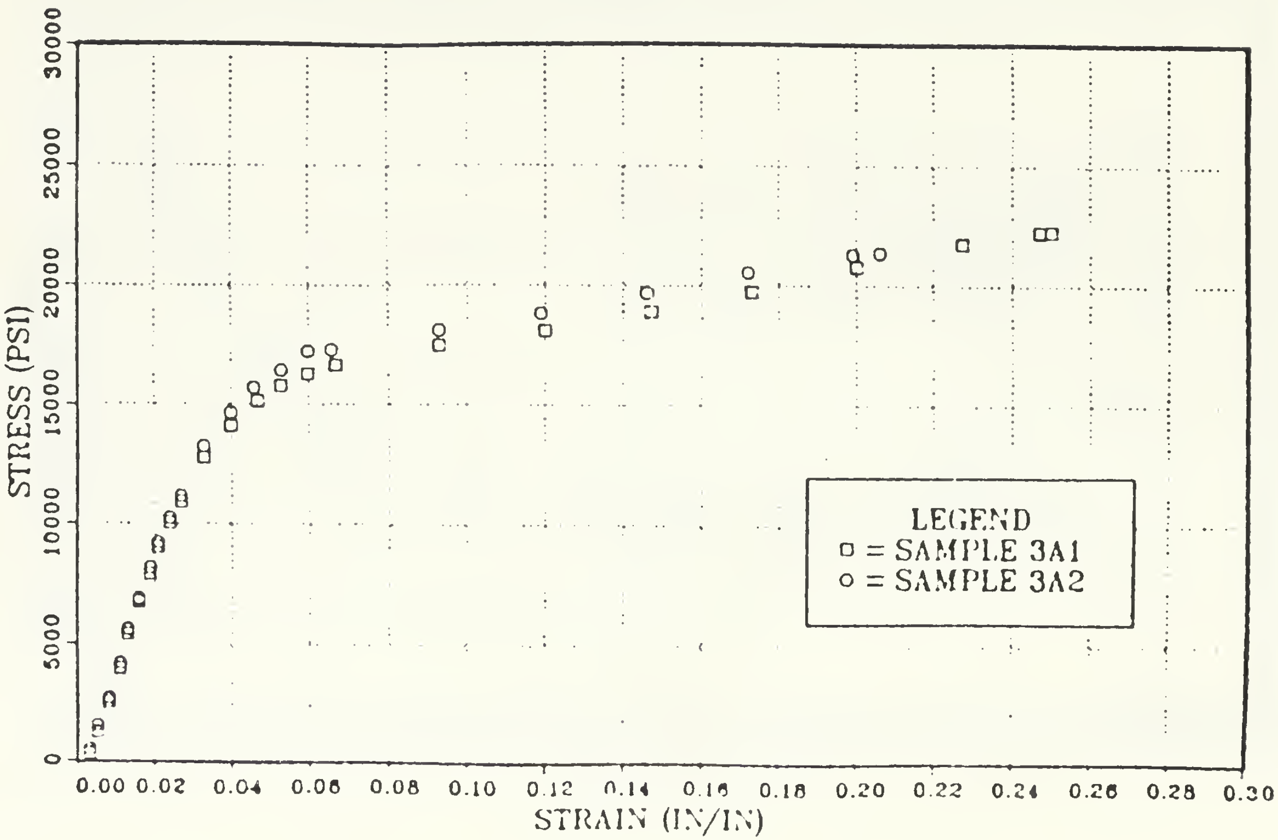

Figure 25. Stress-Strain Curve, 3x3x1-1/2 Inch Samples

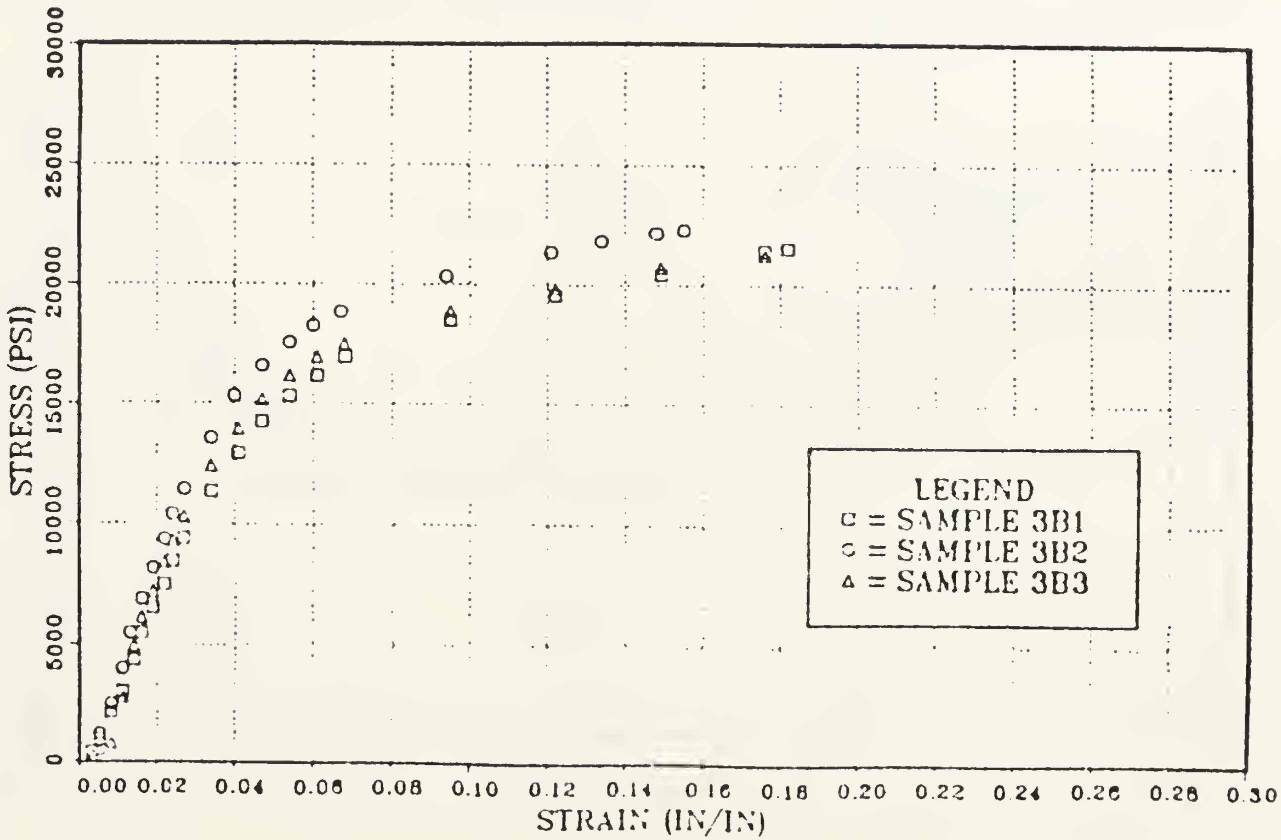

Figure 26. Stress-Strain Curve, 6x6x1-1/2 Inch Samples

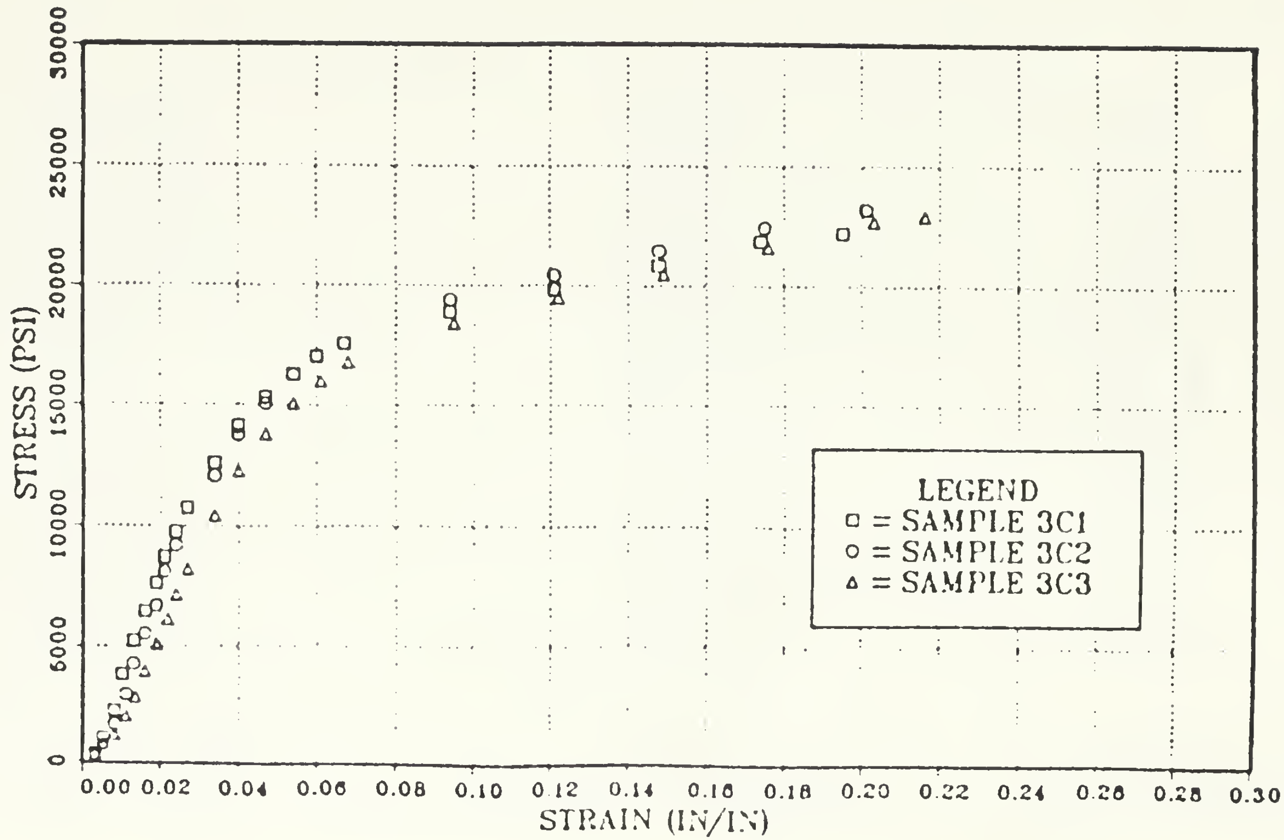

Figure 27. Stress-Strain Curve, 9x9x1-1/2 Inch Samples

Table IX.

Results for Modulus of Elasticity and Compressive Yield Strength

CHOCK SIZE (in x in x in)	MODULUS OF ELASTICITY (psi)	YIELD STRENGTH (psi)
3x3x1/2	323,000	15,560
6x6x1/2	320,000	16,600
9x9x1/2	375,000	16,800
3x3x1	425,000	15,400
6x6x1	420,000	17,680
9x9x1	490,000	17.250
3x3x1-1/2	475,000	17,300
6x6x1-1/2	455,000	17,550
9x9x1-1/2	428,000	17,300

modulus occurred between the 1/2 inch thick chocks and the thicker chocks; however, the degree of exotherm seemed to have very little effect.

The compressive yield strength showed slight increases with higher exotherms. The 1/2 inch thick specimens and the 3x3x1 inch specimens experienced very little exotherm during the curing process and the resultant yield strength in all cases was less than 17,000 psi. The remainder of the 1 inch thick and 1-1/2 inch thick specimens, all of which experienced an exponential temperature rise, had yield strengths in excess of 17,000 psi. Clearly from these results, coupled with the results of ultimate strength presented earlier, the major gains from higher exotherms came in the plastic region.

V. CONCLUSIONS AND RECOMMENDATIONS

In a conversation early on with Mr. George Kollock of Philadelphia Resins Corporation, he indicated that the minimum dimension of the mould would control the exothermic reaction and that nothing "exciting" occurred with samples less than 1 inch thick. Results of this research supported these statements. In all cases, samples of 1/2 inch thickness showed no significant temperature rise. Although the varying thickness caused the greatest increases, the increased lateral dimensions played a role in the maximum temperature achieved.

The exothermic reactions observed during the research can be broken down into two distinct areas. The initial time-temperature profile appears linear until the temperature reaches approximately 160^{o}F. At this time, the curing rate increases and temperature rises exponentially until reaching Tmax. This reaction obeys Arrhenius Law, which states that once the threshold energy of a reaction is exceeded, "For every 10 degrees Celsius rise in temperature, the reaction rate doubles." [Ref. 11] Arrhenius' Rate equation can be expressed as:

$$RATE = A \exp [- E/RT]$$

Where A is an arbitrary constant, E is the threshold energy, R is the universal gas constant and T is absolute temperature.

The ultimate compressive strength showed a significant increase with increased exotherms. The 1 inch thick samples, which had the greatest variation in exotherm as a result of the effects the mould size had on curing temperature, ultimate strength varied by 63.0% from the weakest to the strongest specimens. The greatest increases occurred in the plastic region, where the larger exotherms in effect heat treated the material, increasing toughness and providing greater ductility prior to failure.

The results of testing the 1-1/2 inch samples showed higher exotherms, but a slight reduction in the ultimate strengths of the material. Speculation as to the cause of the strength reduction is independent of temperature and related to the reaction rate. As previously mentioned, as temperature increases exponentially, the reaction rate is increasing also. This means that curing occurs much faster, thereby allowing less time for entrapped air and gases to escape. The voids created serve to weaken the strength of the material. A plot of ultimate strength versus maximum curing temperature is shown for all the specimens tested in Figure 28.

Although this research has found that exotherm has a significant effect on the strength of CHOCKFAST ORANGE, there are a number of questions that must still be addressed.

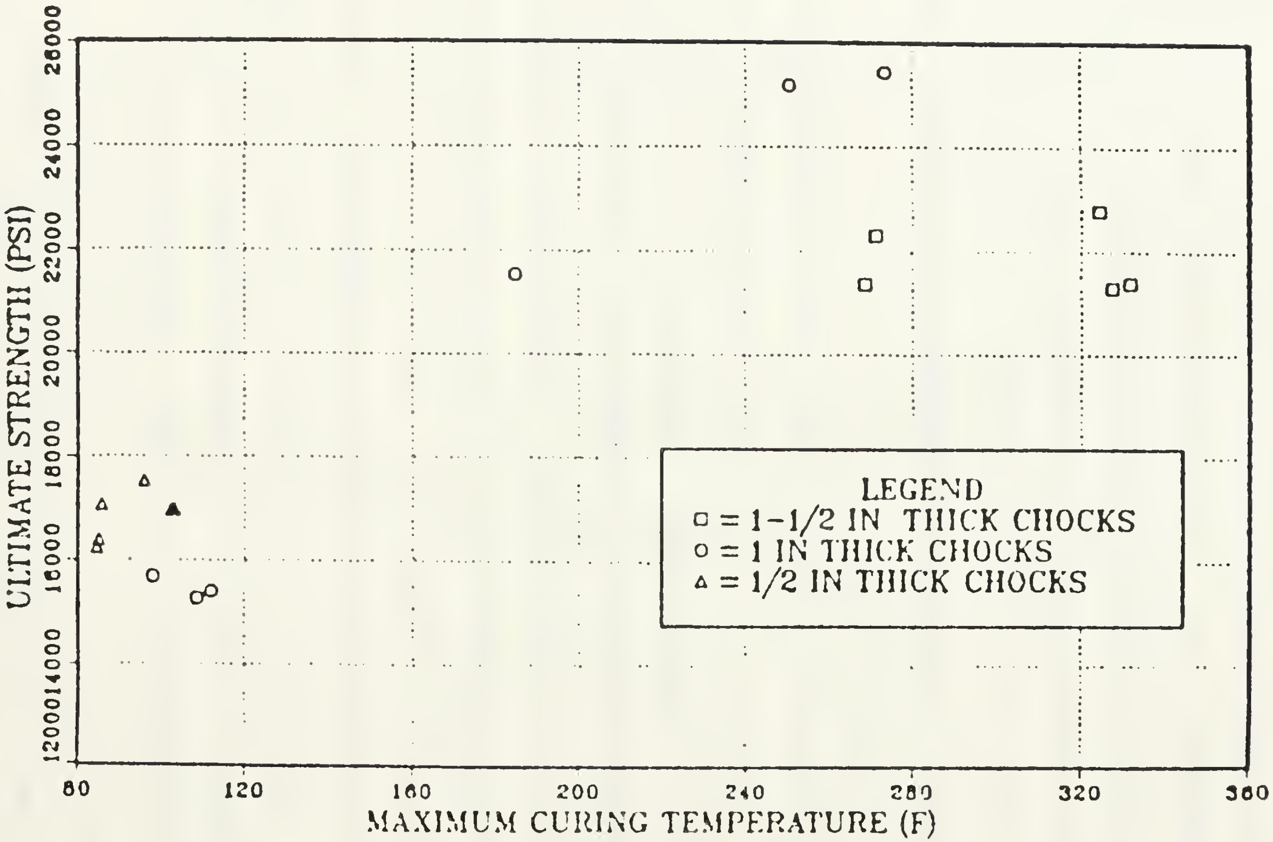

Figure 28. A Plot of Ultimate Strength vs. Maximum Curing Temperatures

During the course of the study, it was discovered that pouring a sample in a mould larger than the sample itself had a large effect on the maximum temperature achieved. This indicates the chemical reaction occurring during the curing process cannot overcome the heat transfer characteristics of the mould. In order to thoroughly define the process that occurs and to be able to accurately predict the extent of the exothermic reaction, further research needs to be accomplished to determine the thermal conductivity of CHOCKFAST ORANGE.

Furthermore, by realizing the extent of the effect of the surrounding environment on the curing process, further research in controlling the environmental conditions should be conducted. Specifically, during this project the maximum strength was achieved with maximum temperatures of 250°F. By controlling the pouring environment, either with external heat sources or cooling, depending on the mass of the compound poured, optimum results should be obtainable.

APPENDIX A

UNCERTAINTY ANALYSIS

ULTIMATE STRENGTH CALCULATIONS

Ultimate stress = ultimate compressive load/initial area

SAMPLE 2A1

A = 3.959 in

P = 60,500 lbf

δP = 125 lbf

$$\frac{\delta A}{A} = \left[\left(\frac{\delta d_1}{d_1}\right)^2 + \left(\frac{\delta d_2}{d_2}\right)^2 \right]^{\frac{1}{2}}$$

d_1 and d_2 represent the initial dimensions of the test specimen. Measurements were made using a .001 inch graduated micrometer. Uncertainty in the measurements was .0005 inches.

$$\frac{\delta A}{A} = \left[\left(\frac{.0005}{2.019}\right)^2 + \left(\frac{.0005}{1.961}\right)^2 \right]$$

δA = .0014 inches

$$\frac{\delta \sigma}{\sigma} = \left[\left(\frac{\delta P}{P}\right)^2 + \left(\frac{\delta A}{A}\right)^2 \right]^{\frac{1}{2}}$$

$$\frac{\delta \sigma}{\sigma} = \left[\left(\frac{125}{60500}\right)^2 + \left(\frac{.0014}{3.959}\right)^2 \right]^{\frac{1}{2}}$$

$\delta\sigma$ = 32 psi

σ = 15,281 $\pm$ 32 psi

STRAIN CALCULATIONS

Strain = deformation/initial thickness

e = .060 in

δe = .0005 in

time delay in recording .001 in

δe_{total} = .0015 in

l = 1.007 in

δl = .005 in

$$\frac{\delta\varepsilon}{\varepsilon} = \left[\left(\frac{\delta e_t}{e}\right)^2 + \left(\frac{\delta l}{l}\right)^2\right]^{\frac{1}{2}}$$

$$\frac{\delta\varepsilon}{\varepsilon} = \left[\left(\frac{.0015}{.060}\right)^2 + \left(\frac{.0005}{1.007}\right)^2\right]^{\frac{1}{2}}$$

$\delta\varepsilon$ = .001 in

ε = .059 $\pm$ .001 in/in

LIST OF REFERENCES

1. Advanced Marine Enterprises, Inc., State-of-the-Art Summary for Epoxy Resins Chocks, p. 1-5, 1982.

2. Ibid., p. 3-1.

3. Ibid., p. 8-1.

4. Ibid., p. 1-3.

5. Lee, H. and Nelville, K., Handbook of Epoxy Resins, p. 1-1, McGraw-Hill, 1967.

6. Ibid., p. 1-2.

7. Ibid., p. 5-1.

8. Ibid., pp. 6-3 and 6-4.

9. Advanced Marine Enterpises, Inc., State-of-the-Art Summary for Epoxy Resins Chocks, pp. 4-1 and 4-2.

10. Ibid., Appendix C, p. 9.

11. Lee, H. and Nelville, K., Handbook of Epoxy Resins, p. 17-8, McGraw-Hill, 1967.

INITIAL DISTRIBUTION LIST

		No. Copies
1.	Defense Technical Information Center Cameron Station Alexandria, Virginia 22314	2
2.	Library, Code 0142 Naval Postgraduate School Monterey, California 93943	2
3.	Chairman, Code 69Mx Department of Mechanical Engineering Naval Postgraduate School Monterey, California 93943	1
4.	Professor David Salinas, Code 69Zc Naval Postgraduate School Monterey, California 93943	2
5.	Naval Sea Systems Command Attn: Mr. William Siekierka Code 55Y3 Department of the Navy Washington, DC 20362	2
6.	David Taylor Naval Ship R and D Center Attn: Mr. Ron Purcell Code 1730.2 Bethesda, Maryland 20084	1
7.	David Taylor Naval Ship R and D Center Attn: Mr. Owen Ritter Code 1211 Bethesda, Maryland 20084	1
8.	LT John D. Stalnaker, USN 1059 Northwood Drive Nappanee, Indiana 46550	2

Stalnaker
The effect of mould size on the exothermic reaction and compressive strength of epoxy resin chocks.

Thesis
S6722
c.1

Stalnaker
The effect of mould size on the exothermic reaction and compressive strength of epoxy resin chocks.